



**U.S. Army
Environmental
Center**

Final Site Safety and Health Plan for Phase II RCRA Facility Investigation Fort Benjamin Harrison Marion County, Indiana

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May 1996

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**Final Quality Assurance
Project Plan for Phase II
RCRA Facility Investigation
Fort Benjamin Harrison
Marion County, Indiana**

Prepared for

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May 15, 1996



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**FINAL QUALITY ASSURANCE PROJECT PLAN
FOR PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON
MARION COUNTY, INDIANA
May 15, 1996**

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1.0 INTRODUCTION

The U.S. Environmental Protection Agency (EPA) requires that all environmental monitoring and measurement efforts mandated or supported by EPA participate in a centrally managed Quality Assurance (QA) program.

Any party generating data under the centrally managed QA program described in this plan has the responsibility to implement minimum procedures to ensure that the precision, accuracy, representativeness, completeness, and comparability of its data are known and documented. To ensure that responsibility is met uniformly, each party must prepare a written QA Project Plan (QAPjP) covering each project the party is to perform.

This QAPjP presents the organization, objectives, functional activities, and specific QA activities and quality control (QC) performance criteria associated with the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) for Fort Benjamin Harrison (FBH), Marion County, Indiana. This QAPjP also describes the specific protocols that will be followed for sampling, sample handling and storage, chain of custody, and field and laboratory analysis.

QA/QC procedures presented in this QAPjP are in accordance with applicable professional technical standards, EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPjP is prepared by HLA to fulfill the requirements of the USAEC under the Total Environmental Program Support (TEPS) Contract DAA15-91-D-0013 in accordance with EPA QAPjP guidance documents, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80) (EPA, 1986), and the Region V Model QAPjP (EPA, 1991c).

1.1 Site History and Background Information

This section describes the general history as well as past and current environmental investigation (EI) activities at FBH.

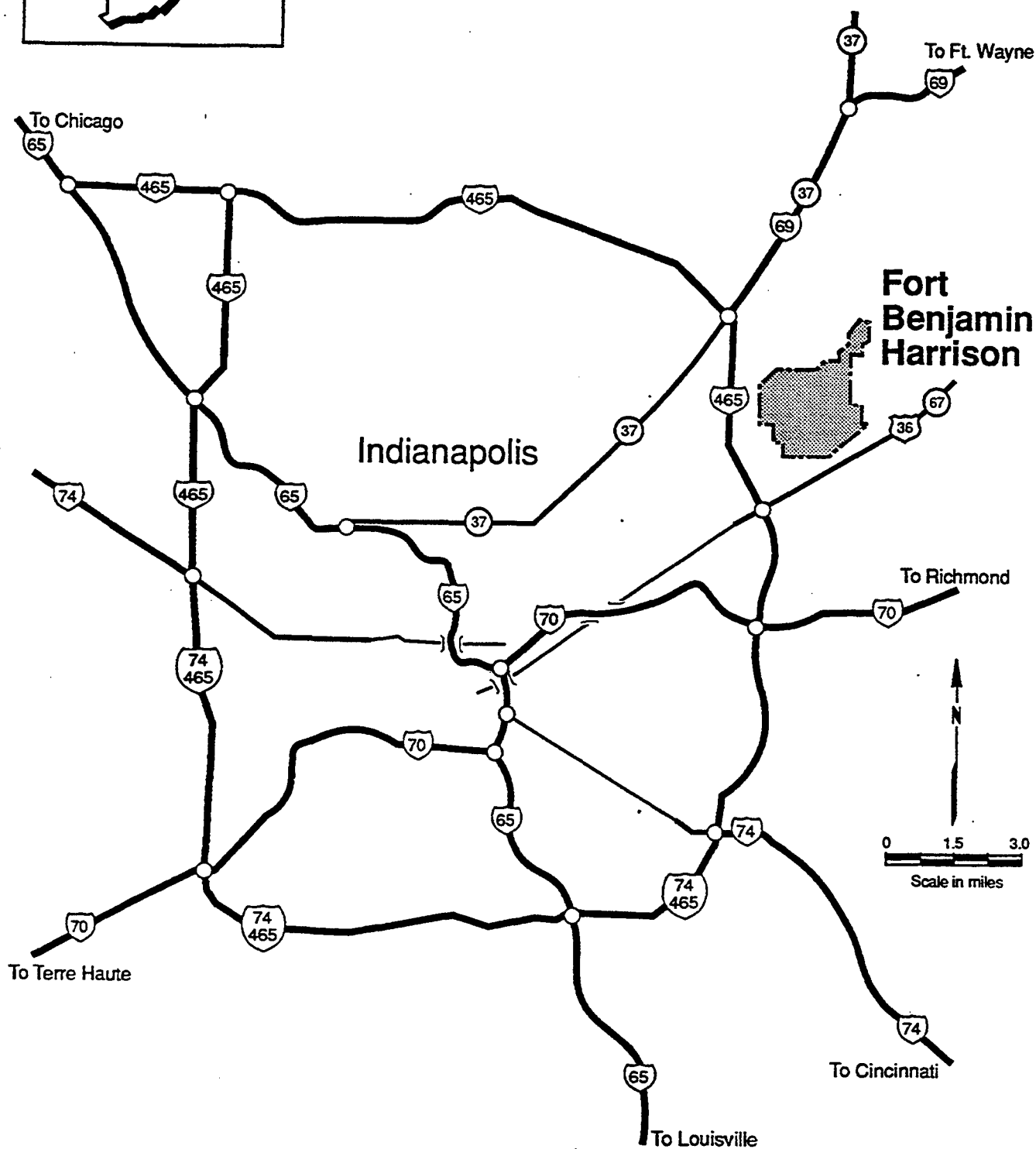
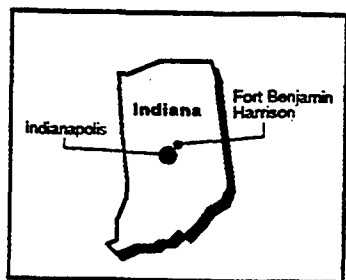
1.1.1 Location

FBH is a U.S. Department of the Army (Army) installation located within Lawrence Township, Marion County, in Central Indiana. The installation is approximately 12 miles northeast of downtown Indianapolis, as shown in Figure 1.1.

1.1.2 Past Data Collection Activities and Current Status

In September 1987, the U.S. Army Soldier Support Center (USASSC) at FBH submitted an application for a RCRA permit to operate a storage facility at the Defense Reutilization and Marketing Office (DRMO). As a result of this application, a RCRA Facility Assessment (RFA) was performed by EPA (EPA, 1991b). The RFA that was conducted identified several solid waste management units (SWMUs) at FBH. The RCRA permit (EPA identification No. IN4 210 090 003) was issued during September 1991, in part by EPA and in part by Indiana Department of Environmental Management (IDEM) (EPA, 1991a). The RCRA permit identified seven SWMUs requiring corrective action. These SWMUs and their potential environmental concerns are described in Table 1.1. USASSC was directed to perform an RFI as a result of these identified SWMUs.

The RFI at FBH is being conducted in two phases to address suspected and actual releases of potentially hazardous materials. Results of the completed Phase I (release assessment) fieldwork and analytical data for each SWMU were used to: (1) evaluate whether a release of potentially hazardous materials has occurred, (2) evaluate which SWMUs contain chemical concentrations presenting a possible threat to human health and the environment, and (3) provide a basis on which to recommend further action, if necessary, during Phase II. Phase II (release characterization) fieldwork and analytical data will be used to (1) better characterize the nature and extent of contamination at each SWMU, where necessary, for which the Phase I data indicate a potential threat to human health and the environment and (2) support the baseline risk assessment and the Corrective Measures Study (CMS) for those SWMUs.



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Figure 1.1
Site Location Map

Table 1.1: Solid Waste Management Units Identified for Investigation and Corrective Action in the RCRA Permit

SWMU Number	Location/Other	SWMU Name	Release/Media	Objective(s) of RFI Release Assessment
FBH2	123rd ARCOM, AMSA (Building 127)	POL Waste Storage Area Motor Pool Maintenance Shop (Container Storage)	Possible spillage of waste oil, antifreeze, and neutralized battery acid to the soil	Assess release(s) to soil
FBH6	DIS (Building 422)	POL Waste Storage Area Heavy Equipment Maintenance (Container Storage)	Spillage of waste oil, gasoline, diesel fuel, spent antifreeze to the soil	Assess release(s) to soil
FBH7*	Auto Craft Shop (Building 705)	Auto Craft Shop Waste Oil Storage Tank (Underground Storage Tank)	Spillage of waste motor oils to the soil	Assess release(s) to soil
FBH8	Proposed Learning Center Site	Former DPDO Container Storage	Unknown releases to the soil	Assess release(s) to soil
FBH10*	(West side of base)	Sanitary Landfill	Leachate seeps have occurred in the past	Assess release(s) to soil, groundwater
FBH11	Abandoned Sewage Treatment Plant	Fire Training Area	Unknown	Assess release(s) to soil, groundwater
FBH17	(East side of base)	Abandoned Incinerator	Unknown (waste disposal is unknown)	Assess release(s) to soil

Source: U.S. Environmental Protection Agency, 1991a.

ARCOM Army Reserve Command
AMSA Area Maintenance Support Activity
DIS Directorate of Installation Support
DPDO Defense Property Disposal Office (now Defense Reutilization and Marketing Office)
POL Petroleum, oil, and lubricants

* Not included in the Phase I Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI); addressed as a part of separate State of Indiana environmental programs

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Introduction

The Phase I RFI at FBH involved the investigation of five SWMUs according to the approved Phase I RFI Technical Sampling Plan (TSP) (HLA, 1993b). The other two SWMUs were addressed under separate State of Indiana environmental programs and have not been included in the RFI. The waste oil tank located at the Auto Craft Shop (Building 705) was closed and removed under the IDEM Underground Storage Tank Program, and the remainder of the site is included in the Phase I EI being performed by the Army as part of the closure of FBH. Environmental concerns at the former sanitary landfill (west side of the base) were addressed by the U.S. Army Corps of Engineers (ACE) under the IDEM Solid Waste Program.

Five SWMUs and additional background sampling locations were identified for investigation as part of the Phase I RFI. The locations of these SWMUs are presented in Figure 1.2. The field sampling program for the Phase I RFI consisted of the following major elements:

- Soil-gas surveys
- Polychlorinated biphenyl (PCB) screening (surface soil)
- Geophysical surveys
- Surface soil sampling
- Soil borings and subsurface-soil sampling
- Monitoring well installation and groundwater sampling
- Surface-water sampling
- Sediment sampling

Results of the Phase I RFI Field program were provided in the Final Phase I RFI Report (HLA, 1994 as revised 1995).

A summary of the Phase I RCRA Facility Investigation, including the intended investigative objective, the medium sampled (e.g., soil), the number of samples collected, the chemical analyses performed, and a brief synopsis of the investigation results, is provided in Table 1.2. The Final Phase I RFI

Introduction

Report was prepared by Harding Lawson Associates (HLA) at the direction of the U.S. Army Environmental Center (USAEC) for the sole use of USAEC and the members of the FBH Base Realignment and Closure (BRAC) Cleanup Team, including the Army, EPA Region V, and the IDEM, the only intended beneficiaries of this work. No other party should rely on the information contained in the report without prior written consent of HLA.

Data obtained during Phase I were used to assess whether a release(s) had occurred at each SWMU and to evaluate the nature of the release(s). Investigative sample analytical results were compared to background concentrations and screening risk criteria during a screening risk evaluation. At SWMUs where no release of hazardous waste or hazardous constituents occurred or where the screening risk evaluation indicated that the release did not pose a potential threat to human health or the environment, no further action was recommended. Additional activities (including the Phase II RFI, baseline risk assessment, and CMS, as appropriate) were recommended at SWMUs where a release has occurred that may pose a threat to human health or the environment.

Basewide conclusions for the Phase I RFI are summarized below. Conclusions related to individual SWMUs are summarized in Table 1.3.

The evaluation background media (e.g., soil, sediment, groundwater, and surface water) revealed three areas that may need to be addressed during future phases of investigation:

- Background metals concentrations in soil and sediment at FBH do not appear to have been adequately characterized. Background analysis failed to screen out metals in soil at several sites where the metals are likely to be natural and unrelated to activities at the SWMUs.
- Polynuclear aromatic hydrocarbon (PAH) concentrations in background and investigative samples were occasionally found to be higher than those that would normally be expected, and are believed to be present because of scattered coal fragments left over from past coal storage activities in some areas.
- Pesticides such as DDT, DDD, DDE, dieldrin, and chlordane were detected in many surface soil samples collected basewide, including background samples. This wide distribution of pesticides may be indicative of historical basewide pesticide application and may not be related to SWMU-specific activities.

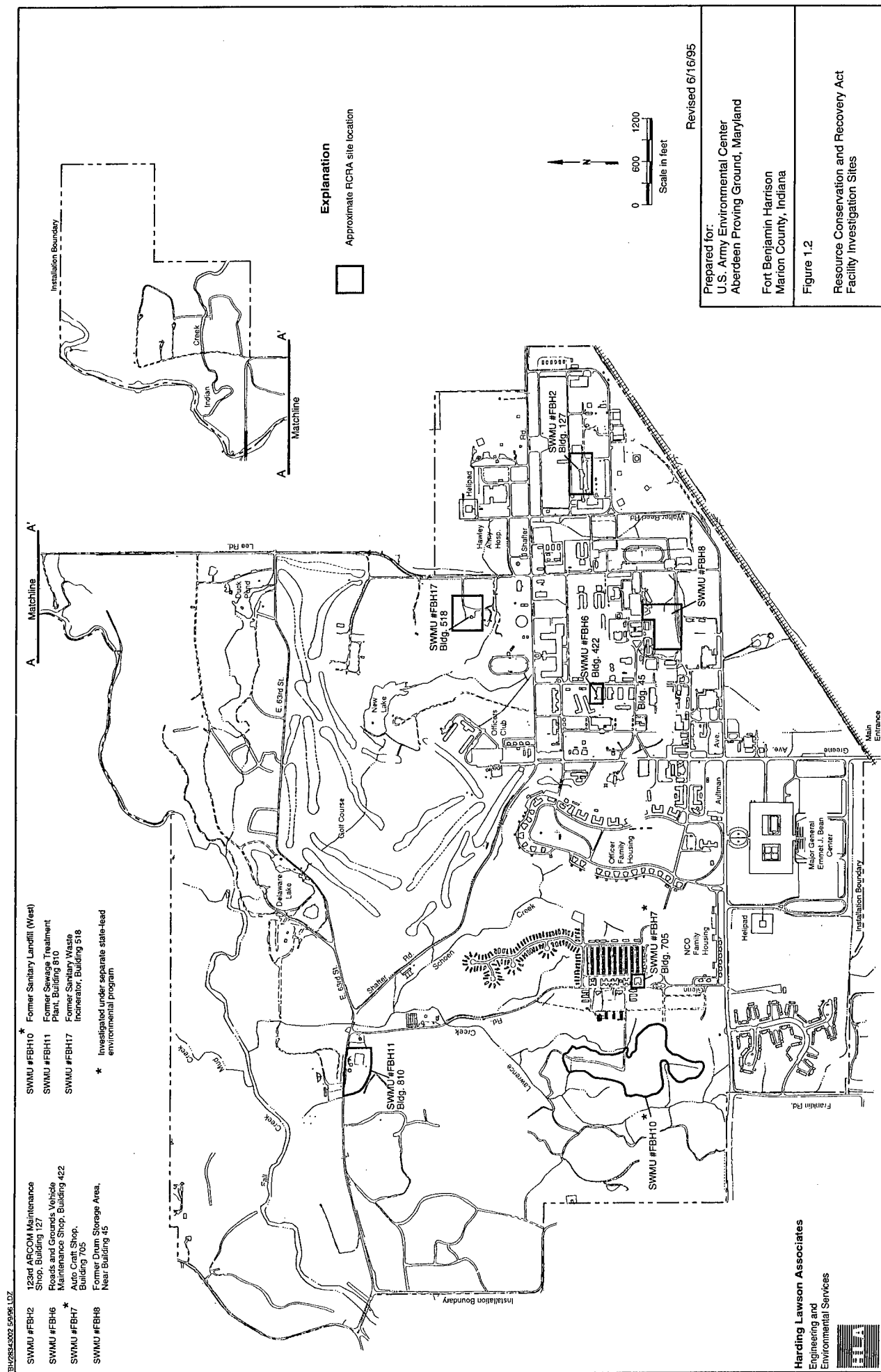


Table 1.2: Summary of Phase I RCRA Facility Investigation

Site Identification	RFI Objective	Investigation Activities Performed			Summary of Phase I Results
		Field Activity	Number of Samples/Locations	Chemical Analyses	
SWMU #FBH2 123rd ARCOM Maintenance Shop, Building 127	Assess release(s) to soil	Surface-soil sampling	2	S,T,M	Target analyte concentrations did not exceed background values.
		Subsurface-soil sampling	11 from 5 borings	V,S,T,M	Metals, cyanide, and selenium were detected above background concentrations, but below screening risk criteria. PAH concentrations exceeding background values for many PAHs and screening risk criteria for 4 PAHs.
		Soil-gas sampling	11	V	Solvents, chloroform, 1,1,1-TCA, TCE, PCE, toluene, and total volatile hydrocarbons were detected.
SWMU #FBH6 Roads and Grounds Vehicle Maintenance Shop, Building 422	Assess release(s) to soil	Subsurface-soil sampling	24 from 5 borings	V,T,T,M	Thirteen metals were detected above background concentrations; of these, maximum detections of beryllium and thallium exceed screening risk criteria. Acetone was detected in one sample below the screening risk criteria.
		Groundwater sampling	4 samples, 1 sample from each of 4 monitoring wells	V,T	Acetone was detected in one sample but is suspected to be a laboratory artifact; however, the reported concentration exceeds the screening risk criteria.
		Soil-gas sampling	---	---	Site conditions did not allow collection of soil-gas samples.
SWMU #FBH8 Former Drum Storage Area Southeast of Building 45	Assess release(s) to soil	Surface-soil sampling	18	PCB field screening	PCBs were detected at one location.
			12	S,T,M,P,D	PCBs, dioxins, and furans were detected above background concentrations; only PCB-1260 exceeded the screening risk criteria for human health. Dioxins and furans exceeded screening environmental hazard criteria.

Table 1.2 (continued)

Site Identification	RFI Objective	Investigation Activities Performed			Summary of Phase I Results
		Field Activity	Number of Samples/Locations	Chemical Analyses	
SWMU #FBH8 Former Drum Storage Area Southeast of Building 45 (continued)		Subsurface-soil sampling	19 from 8 borings	V,S,TM,P,D	Thirteen metals/inorganics, PCBs, dioxins, and furans were detected above background concentrations. Arsenic, cobalt, dioxins, furans, and PCB-1260 were detected above screening risk criteria.
		Groundwater sampling	4 samples, 1 sample from each of 4 monitoring wells	V,S,DM,TM,P	Sixteen metals and six organics were detected above background concentrations. Manganese and TCE were detected above screening risk criteria.
	Assess release(s) to soil, groundwater	Surface-soil sampling	10	S,TM,P,H,AN	Four metals and fifteen organics were detected above background concentrations. No analytes were detected above screening risk criteria.
SWMU #FBH11 Former Sewage Treatment Plant, Building 810		Geophysical survey	---	---	Two metallic anomalies were identified.
		Subsurface-soil sampling	17 from 10 borings	V,S,TM,P,H,AN	Fourteen metals/inorganics, pesticides, PCBs, PAHs, and solvents were detected above background concentrations. Arsenic, beryllium, lead, PAHs, and thallium were detected above screening risk criteria.
		Groundwater sampling	5 samples, 1 from each of 5 monitoring wells	V,S,TM,DM,P,H,AN	Seventeen metals/inorganics and five organics were detected above background concentrations. Chromium, manganese, and vanadium were detected above screening risk criteria.
		Sediment sampling	4	V,S,TM,P,H,AN	Fifteen metals/inorganics, four pesticides, and eight PAHs/other inorganics were detected above background concentrations. Arsenic was detected above soil screening risk criteria.

Table 1.2 (continued)

Site Identification	RFI Objective	Investigation Activities Performed			Summary of Phase I Results
		Field Activity	Number of Samples/Locations	Chemical Analyses	
SWMU #FBH17 Former Sanitary Waste Incinerator Building 518	Assess release(s) to soil	Surface-soil sampling	25	S,TM,P,H	Thirteen metals/inorganics, three pesticides, PCBs, and PAHs were detected above background concentrations. Aluminum, arsenic, PAHs, PCB-1260, and thallium were detected above screening risk criteria.
		Geophysical survey	---	---	Several metallic anomalies were identified.
		Subsurface-soil sampling (native soil)	12	V,S,TM,P,H	Three metals and four PAHs were detected above background concentrations. No analytes were detected above screening risk criteria.
		Subsurface-soil sampling (ash fill area)	12	V,S,TM,P,H	Five metals and five PAHs were detected above background concentrations. Beryllium, PAHs, copper, and lead were detected above screening risk criteria.
Background Sampling	Assess background concentrations in soil, groundwater	Surface-soil sampling	21	S,TM,P,H,AN,L	Background soil and groundwater samples were used to establish background concentrations for the installation.
		Subsurface-soil sampling	49	V,S,TM,P,H,L	
		Groundwater sampling	17	V,S,TM,DM,P,H,L	
Basewide Sampling	Assess release(s) to surface water, sediment	Surface-water sampling	15	V,S,TM,DM,P,H,AN	These samples were used to assess potential source areas to surface water and sediment.
		Sediment sampling	19	V,S,TM,P,H,AN	

Table 1.2 (continued)

1,1,1-TCA	1,1,1-Trichloroethane
AN	Ammonia and nitrate
ARCOM	123rd Army Reserve Command
D	High-resolution dioxins and furans
DM	Dissolved metals
H	Herbicides
L	Landfill parameters
P	Pesticides/PCBs
PAH	Polynuclear aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PCE	Tetrachloroethene
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
S	Semivolatile organic compounds
SWMU	Solid Waste Management Unit
T	Total petroleum hydrocarbons
TCE	Trichloroethene
TM	Total metals
V	Volatile organic compounds

Table 1.3: Summary of Phase I RCRA Facility Investigation Conclusions and Recommendations

SWMU	Medium	Chemicals of Concern	Screening Risk		Screening Environmental Hazard		Recommendations
			Criteria Exceeded?		Criteria Exceeded?		
SWMU #FBH2 123rd ARCOM Maintenance Shop, Building 127	Surface soil	None	NP	No	No	No further action	Conduct Phase II subsurface soil sampling near former POL storage area.
	Subsurface soil	PAHs	Yes	No	No	No further action	
SWMU #FBH6 Roads and Grounds Vehicle Maintenance Shop, Building 422	Subsurface soil	None	No ^a	No	No	No further action	Conduct an additional round of groundwater sampling.
	Groundwater	Acetone	Yes	NP	NP	Conduct an additional round of groundwater sampling.	
SWMU #FBH8 Former Drum Storage Area Southeast of Building 45	Surface soil	Dioxins ^b , furans ^b , PCB-1260	Yes ^a	Yes	Yes	Conduct Phase II surface soil sampling to further assess extent of contamination. Conduct a baseline risk assessment and ecological assessment.	Conduct Phase II subsurface soil sampling to further assess extent of contamination. Conduct a baseline risk assessment and ecological assessment.
	Subsurface soil	Arsenic, cobalt ^b , dioxins ^b , furans ^b , PCB-1260	Yes ^a	Yes	Yes	Conduct Phase II subsurface soil sampling to further assess extent of contamination. Conduct a baseline risk assessment and ecological assessment.	
	Groundwater	Manganese, TCE	Yes	NP	NP	Conduct an additional round of groundwater sampling. Conduct a baseline risk assessment and ecological assessment.	
SWMU #FBH11 Former Sewage Treatment Plant, Building 810	Surface soil	None	No ^c	No	No	No further action	Conduct Phase II soil sampling, and conduct trenching/excavation in the area of geophysical anomalies to identify the nature of the anomalies; conduct a baseline risk assessment and ecological assessment.
	Subsurface soil	Arsenic, beryllium, lead ^b , PAHs, thallium, PCB-1260	Yes	Yes	Yes	Conduct Phase II soil sampling, and conduct trenching/excavation in the area of geophysical anomalies to identify the nature of the anomalies; conduct a baseline risk assessment and ecological assessment.	
	Groundwater	Chromium, manganese, vanadium	Yes	NP	NP	Conduct an additional round of groundwater sampling; conduct a baseline risk assessment and ecological assessment.	
	Sediment	Arsenic ^d	NP	NP	NP	No further action	

Table 1.3 (continued)

SWMU	Medium	Chemicals of Concern	Screening Risk Criteria Exceeded?	Screening Environmental Hazard Criteria Exceeded?	Recommendations
SWMU #FBH17 Former Sanitary Waste Incinerator Building 518	Surface soil	Arsenic, copper ^b , PAHs	Yes ^c	Yes	Conduct Phase II surface soil sampling to further assess extent of contamination. Conduct a baseline risk assessment and ecological assessment. Conduct a CMS based on the outcome of the baseline risk assessment.
	Subsurface soil (native soil)	None	No ^a	NP	No further action
	Subsurface soil (ash area)	Aluminum ^b , cobalt ^b , lead ^b	No ^c	Yes	Conduct trenching/excavation in the area of geophysical anomalies to identify the nature of the anomalies. Conduct a baseline risk assessment and ecological assessment. Conduct a CMS based on the outcome of the baseline risk assessment.
	Groundwater	---	---	---	Install monitoring wells and collect groundwater samples to assess possible groundwater contamination.

--- Not yet evaluated

> Greater than

CMS Corrective Measures Study

DPDO Defense Property Disposal Office

NP Not performed

PAH Polynuclear aromatic hydrocarbon

PCB Polychlorinated biphenyl

POL Petroleum, oil, and lubricants

RCRA Resource Conservation and Recovery Act

SWMU Solid Waste Management Unit

TCE Trichloroethene

a. After correction for background concentrations.

b. Based on average concentrations at site.

c. Potential chemical of concern for screening environmental hazard evaluation only.

d. Screening evaluations were not performed for sediment; however, this exceedance of soil screening risk criteria by arsenic is not considered sufficient cause for further action.

Excluding the elevated concentrations of PAHs basewide in surface and shallow soil and the higher than background levels of trace metals in surface soil, subsurface soil, and sediment, only small amounts of chemical constituents in environmental media (e.g., soil, sediment, surface water, groundwater) were found. The metals concentrations in soil at most of the SWMUs are not significantly elevated above naturally occurring background values. The exception to this statement is the former incinerator (SWMU #FBH17).

1.2 Project Scope and Objectives

Overall objectives for site investigations are defined in Section 4.1 of the TSP, and site-specific objectives regarding sample selection, frequency, and analyses are defined in Section 5.0 of the TSP (HLA, 1996a). The Phase II RFI site investigation objectives are summarized as follows:

- Provide additional groundwater monitoring data at SWMUs where hazardous waste or hazardous constituents were identified.
- Provide additional data to evaluate the presence of potentially hazardous waste or hazardous constituents at sites where Phase I data were inconclusive.
- Further evaluate the nature and extent of potentially hazardous waste or hazardous constituents.
- Collect additional data to support a baseline risk assessment.
- Collect additional data to support a CMS.
- Collect additional data to support property transfer for base closure at FBH.

Data obtained during the Phase II RFI will be used to complete the RFI Report and to support the CMS and baseline risk assessment, if required by EPA.

1.3 Sample Network Design and Rationale

The sample network design and rationale for sample locations (in respective media) are described in detail in Section 5.0 of the TSP (HLA, 1996a).

1.4 Parameters to be Tested and Frequency

Sample matrices, analytical parameters, and frequencies of sample collections are presented in Sections 5.0 and 6.0 of the TSP. Analytical methods that will be used to analyze Phase II RFI samples are discussed in Section 7.0 of this QAPjP.

1.5 Intended Data Usage and Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality of information required to support decisions made during Phase II RFI activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels that address various data uses and the QA/QC effort and methods required to achieve the desired level of quality (EPA, 1987). These levels are as follows:

- Screening (DQO Level I): This level provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, initial site characterization to locate areas for subsequent and more accurate analysis, and engineering screening of possible remedial alternatives (bench-scale tests).
- Field Analyses (DQO Level II): This level provides rapid results and better data quality than Level I. This level may include mobile laboratory-generated data depending on the level of QC exercised.
- Engineering (DQO Level III): This level provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory-generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround times used for screening purposes but without full QC documentation).
- Confirmation (DQO Level IV): This level provides the highest level of data quality and is used for purposes of risk assessment and evaluation of remedial alternatives. These analyses require the highest level of analytical and data validation procedures in accordance with EPA-recognized protocol.
- Nonstandard (DQO Level V): This level refers to analyses by nonstandard protocols (e.g., when determining detection limits or when analysis of a nonconventional parameter is required). These analyses often require method development or adaptation. This level of QC is usually similar to Level IV data.

Because of the intended multiple uses of the data to be collected, intended data uses are discussed separately for each medium. Details of the analytical methods to be used for the investigation are presented in Section 7.0.

1.5.1 Specific Objectives and Associated Tasks

Media-specific data quality objectives for the RFI, based on intended data uses and appropriate analytical levels (EPA, 1991c), are described in the following subsections. In addition, Table 1.4 provides a summary of Phase II RFI sample collection activities. The table also includes a description of the type of sample analyses to be performed, intended data use, data quality level, and list of potential health-based target levels. Health-based target levels are included for reference and are not intended to convey or imply specific screening or action levels for the sites. However, regulatory levels to evaluate concentrations of constituents in site-specific media have not been selected pending EPA and IDEM review.

1.5.1.1 Groundwater

Data collected from the chemical analysis of RFI groundwater samples will be used for site characterization, risk assessment, and evaluation of remedial alternatives. The highest priority data use is the risk assessment; therefore, analytical Level IV is considered to be the most appropriate level of quality for groundwater. Groundwater analyses for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), pesticides/PCBs, herbicides, metals, and dioxins/furans will be Level IV data. Groundwater analyses for total organic carbon (TOC) will be Level III data. The Level III data will not be used for risk assessment.

1.5.1.2 Soil

Data collected from the chemical analysis of RFI soil samples will be used for site characterization, risk assessment, and evaluation of remedial alternatives. The highest priority data use is the risk assessment; therefore, analytical Level IV is considered to be the most appropriate level of quality for soil analyses. Soil analyses for VOCs, SVOCs, pesticides/PCBs, herbicides, and dioxins/furans will be Level IV data. Soil analyses for TOC, total petroleum hydrocarbons (TPH), and cation exchange capacity (CEC) will be Level III. The Level III data will not be used for risk assessment.

1.5.1.3 Polychlorinated Biphenyl Soil Screening

Data collected from screening the soil for PCBs will be used for site characterization. Because the PCB soil screening will occur onsite, analytical Level II is considered to be the most appropriate level of quality for the PCB soil screening. Onsite soil screening for PCBs allows for rapid analyses of soil samples. Results of the PCB soil screening can be used to select additional sampling locations while the field sampling crew is still mobilized onsite. The Level II data will not be used for risk assessment.

1.5.1.4 Volatile Organic Compounds Screening

Data collected from screening of surface-soil samples for VOCs will be used as criteria for selecting surface-soil samples for laboratory VOC analyses. Because the VOC screening will occur onsite, and provide primarily qualitative analyses, analytical Level I is considered to be the most appropriate and will be used for the VOC soil screening. The Level I data will not be used for risk assessment.

1.5.1.5 Physical Analyses

Physical testing will be performed on 20 percent of the soil samples collected to assess the accuracy and consistency of the geologist field descriptions of surface and subsurface soil. Testing will include Atterberg limits (American Society for Testing and Materials [ASTM] D-4315), grain-size distribution (ASTM D-422), and Minus 200 Test, percentage of silt and clay (ASTM D-1140). The physical analysis of the soil will be Level III. The Level III data will not be used for risk assessment.

1.6 Project Schedule

The project schedule for the Phase II RFI at FBH is presented in Figure 1.3. Fieldwork is scheduled to begin June 16, 1996, subject to approval of the Work Plan by EPA and IDEM by June 15, 1996. The Final RFI report is scheduled to be completed by March 24, 1997.

Table 1.4: Data Quality Objectives Summary Table

Site Identification/Activity	Analyses ^a	Data Use	Data Quality Level ^b	Health-Based Target Levels ^c
SWMU #FBH2 123rd ARCOM Maintenance Shop, Building 127				
Subsurface-Soil Sampling	<ul style="list-style-type: none"> • Total Metals • Volatile organic compounds • Semivolatile organic compounds • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs
SWMU #FBH6 Roads and Grounds Vehicle Maintenance Shop, Building 422				
Soil VOC Screening	<ul style="list-style-type: none"> • Volatile organic compounds 	<ul style="list-style-type: none"> • Evaluate extent of contamination 	Level I	---
Surface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Polychlorinated biphenyls • Total petroleum hydrocarbons^d • Cation exchange capacity^d • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs
Subsurface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Polychlorinated biphenyls • Total petroleum hydrocarbons^d • Cation exchange capacity^d • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate nature and extent of contamination • Support risk assessment • Support evaluation of alternatives 	Level III Level IV	DQLs

Table 1.4 (continued)

Site Identification/Activity	Analyses ^a	Data Use	Data Quality Level ^b	Health-Based Target Levels ^c
Groundwater Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total and dissolved metals • Polychlorinated biphenyls • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment 	Level III, Level IV	MCLs DQLs
SWMU #FBH8 Former Drum Storage Area Southeast of Building 45				
Soil PCB Screening	<ul style="list-style-type: none"> • Polychlorinated biphenyls 	<ul style="list-style-type: none"> • Evaluate extent of contamination 	Level II	---
Soil VOC Screening	<ul style="list-style-type: none"> • Volatile organic compounds 	<ul style="list-style-type: none"> • Evaluate extent of contamination 	Level I	---
Surface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds 	<ul style="list-style-type: none"> • Evaluate extent of contamination • Evaluate support risk assessment • Support evaluation of alternatives 	Level IV	MCLs DQLs
Subsurface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides and polychlorinated biphenyls • Dioxins/furans and herbicides • Cation exchange capacity^d • Total organic carbon^d • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate extent of contamination • Support risk assessment • Support evaluation of alternatives 	Level III Level IV	DQLs
Groundwater Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Dissolved metals • Pesticides and polychlorinated biphenyls • Dioxins/furans and herbicides • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment 	Level IV	MCLs DQLs

Table 1.4 (continued)

Site Identification/Activity	Analyses ^a	Data Use	Data Quality Level ^b	Health-Based Target Levels ^c
Soil Trenching	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides and polychlorinated biphenyls • Dioxins/furans and herbicides • Cation exchange capacity^d • Total organic carbon^d • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	MCLs DQLs
SWMU #FBH11 Former Sewage Treatment Plant, Building 810				
Soil VOC Screening	<ul style="list-style-type: none"> • Volatile organic compounds 	<ul style="list-style-type: none"> • Evaluate extent of contamination 	Level I	---
Surface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides • Polychlorinated biphenyls • Herbicides • Cation exchange capacity^d • Total organic carbon^d • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs
Subsurface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides • Polychlorinated biphenyls • Herbicides • Cation exchange capacity^d • Total organic carbon^d • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs

Table 1.4 (continued)

Site Identification/Activity	Analyses ^a	Data Use	Data Quality Level ^b	Health-Based Target Levels ^c
Groundwater Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Dissolved metals • Pesticides • Polychlorinated biphenyls • Herbicides • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs, MCLs
Soil Trenching	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides • Polychlorinated biphenyls • Herbicides • Cation exchange capacity^d • Total organic carbon^d • Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs
SWMU #FBH17 Former Sanitary Waste Incinerator Building 518				
Soil VOC Screening	<ul style="list-style-type: none"> • Volatile organic compounds 	<ul style="list-style-type: none"> • Evaluate extent of contamination 	Level I	---
Surface-Soil Sampling	<ul style="list-style-type: none"> • Volatile organic compounds • Semivolatile organic compounds • Total metals • Pesticides • Polychlorinated biphenyls • Herbicides • Dioxins/furans • Cation exchange capacity^d • Total organic carbon^d 	<ul style="list-style-type: none"> • Evaluate presence or absence of contaminants • Support risk assessment • Support evaluation of alternatives 	Level III, Level IV	DQLs

Table 1.4 (continued)

Site Identification/Activity	Analyses ^a	Data Use	Data Quality Level ^b	Health-Based Target Levels ^c
Subsurface-Soil Sampling	<ul style="list-style-type: none"> Dioxins/furans Cation exchange capacity^d Total organic carbon^d 	<ul style="list-style-type: none"> Evaluate presence or absence of contaminants Support risk assessment Support evaluation of alternatives 	Level III Level IV	DQLs
Groundwater Sampling	<ul style="list-style-type: none"> Volatile organic compounds Semivolatile organic compounds Total metals Dissolved metals Pesticides Polychlorinated biphenyls Herbicides Dioxins/furans Total organic carbon^d 	<ul style="list-style-type: none"> Evaluate presence or absence of contaminants Support risk assessment Support evaluation of alternatives 	Level IV	DQLs, MCLs
Soil Trenching	<ul style="list-style-type: none"> Volatile organic compounds Semivolatile organic compounds Total metals Pesticides Polychlorinated biphenyls Herbicides Dioxins/furans Cation exchange capacity^d Total organic carbon^d Total petroleum hydrocarbons^d 	<ul style="list-style-type: none"> Evaluate presence or absence of contaminants Support risk assessment Support evaluation of alternatives 	Level III Level IV	DQLs

DQL Data Quality Levels
FBH Fort Benjamin Harrison
IDEM Indiana Department of Environmental Management
MCL Maximum Contaminant Level
SWMU Solid Waste Management Unit

Table 1.4 (continued)

VOC Volatile organic compounds

- a. Analyses will be performed using the following analytical methods:

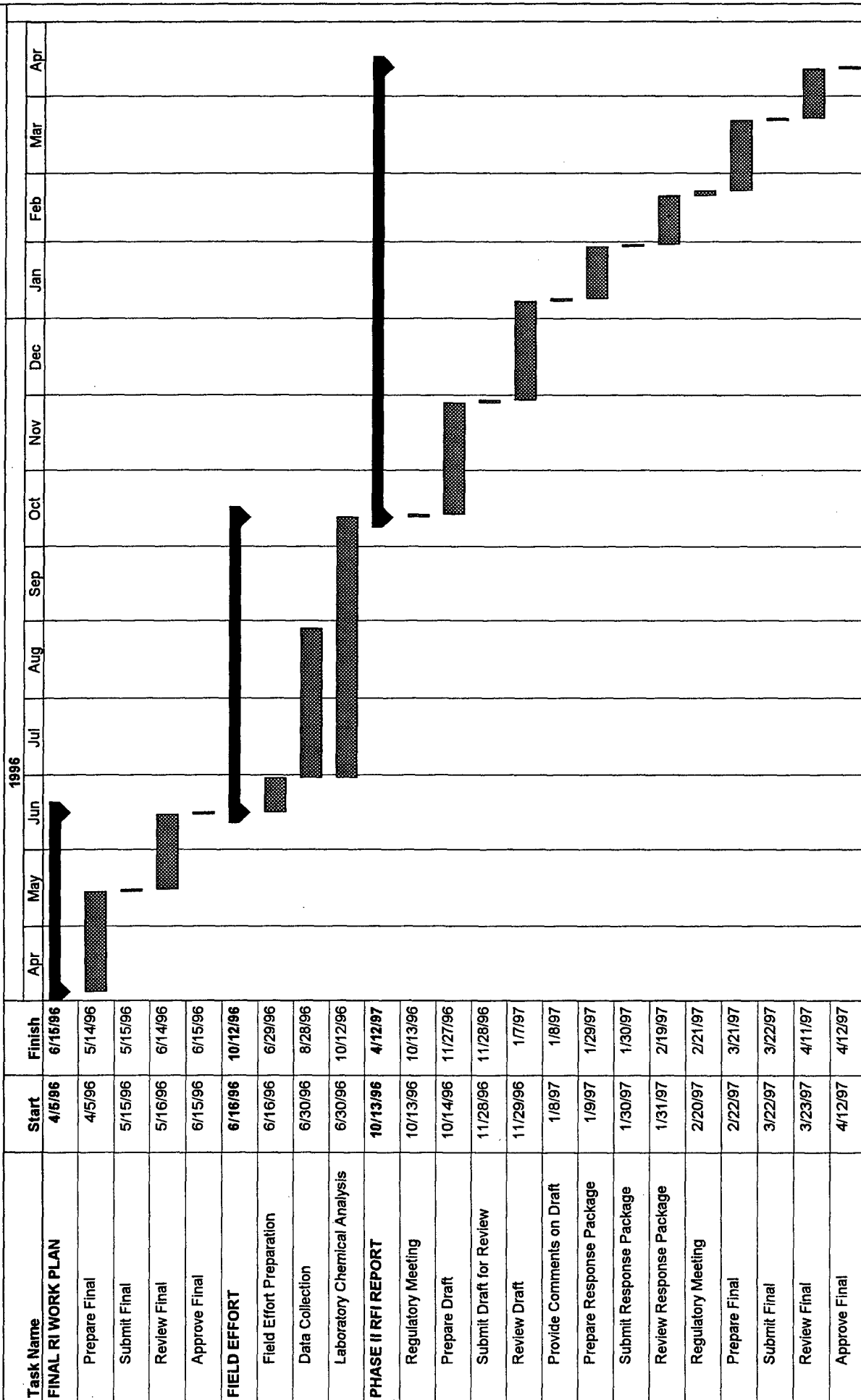
Analytical Parameters	Proposed Analytical Method	
	Water	Soil
Volatile organic compounds	CLP SOW OLC01.0	CLP SOW OLM03.1
Semivolatle organic compounds	CLP SOW OLC01.0	CLP SOW OLM03.1
Pesticides	CLP SOW OLC01.0	CLP SOW OLM03.1
Polychlorinated biphenyls	CLP SOW OLC01.0	CLP SOW OLM03.1
Herbicides	SW-846, 8150*	SW-846, 8150*
Total metals	CLP SOW ILC01.0	CLP SOW ILM03.0
Dissolved metals	CLP SOW ILC01.0	CLP SOW ILM03.0
Total petroleum hydrocarbons	SW-846 Modified 8015*	SW-846 Modified 8015*
Total organic carbon	SW-846 9060*	SW-846 9060*
Dioxins/furans	SW-846 8290*	SW-846 8290*
Cation exchange capacity	---	SW 846 9080*

CLP SOW Contract Laboratory Program Statement of Work

* USEPA 1994, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods SW-846.

- b. DQO Levels as defined in Environmental Protection Agency Region V Model Superfund Quality Assurance Project Plan, May 1991.
Level I Screening Data
Level II Field Analyses
Level III Engineering Data
Level IV Confirmational, Contract Laboratory Program or equivalent
- c. Tentative levels pending regulatory review and comment
- d. Total petroleum hydrocarbon analysis, cation exchange capacity, and total organic carbon will be Level III data.

**FIGURE 1.3 - SCHEDULE FOR PHASE II OF THE
RCRA FACILITY INVESTIGATION**



2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

This section describes the task management and QA organization structure that will be implemented to ensure that the data collected in support of the Phase II RFI meet the project objectives and the specific requirements outlined in this QAPjP.

2.1 Project Organization Chart

The overall task organization chart and list of key personnel responsibilities are provided in the FBH Management/Resource Plan (MRP). The task-specific QA organizational structure is shown in Figure 2.1. In this organizational structure, personnel identified for TEPS-level responsibilities are titled "program" personnel. Personnel identified for the RFI task-level responsibilities are titled "task" personnel. Brief descriptions of the QA responsibilities of key HLA program and task QA personnel and laboratory QA personnel follow.

2.2 Management Responsibilities

Base Realignment and Closure Environmental Cleanup Team - Richard Blume-Weaver, FBH Base Realignment and Closure Environmental Coordinator; Lorraine Wright, IDEM Remedial Program Manager; Karen Mason-Smith, EPA Superfund Remedial Project Manager

- Oversee FBH environmental investigations.
- Review base closure activities.
- Review project planning documents submitted by the Army to EPA Region V and IDEM.
- Review project Phase II RFI reports submitted by the Army to EPA Region V and IDEM.

EPA RCRA Project Coordinator - Gale Hruska

- Overall responsibility for all phases of the FBH RFI.
- Review project planning documents submitted by the Army to EPA Region V.
- Review project Phase II RFI reports submitted by the Army to EPA Region V.
- Direct EPA oversight of the FBH RFI.

Project Organization and Responsibility

USAEC Project Officer - William C. Nelson

- Oversee implementation of the project.
- Commit resources necessary to meet project objectives.
- Assure project technical, financial, and scheduling objectives are met.
- Review planning documents and summary reports submitted by HLA to the Army.
- Act as point of contact and control for the Army with EPA Region V and IDEM.

EPA Region V Superfund Chemist - Denise Boone

- Review and approve QAPjP.
- Conduct external performance and system audits of RFI laboratory.
- Review and evaluate analytical field and laboratory procedures.

IDEM QA/QC Officer - Manuela Johnson

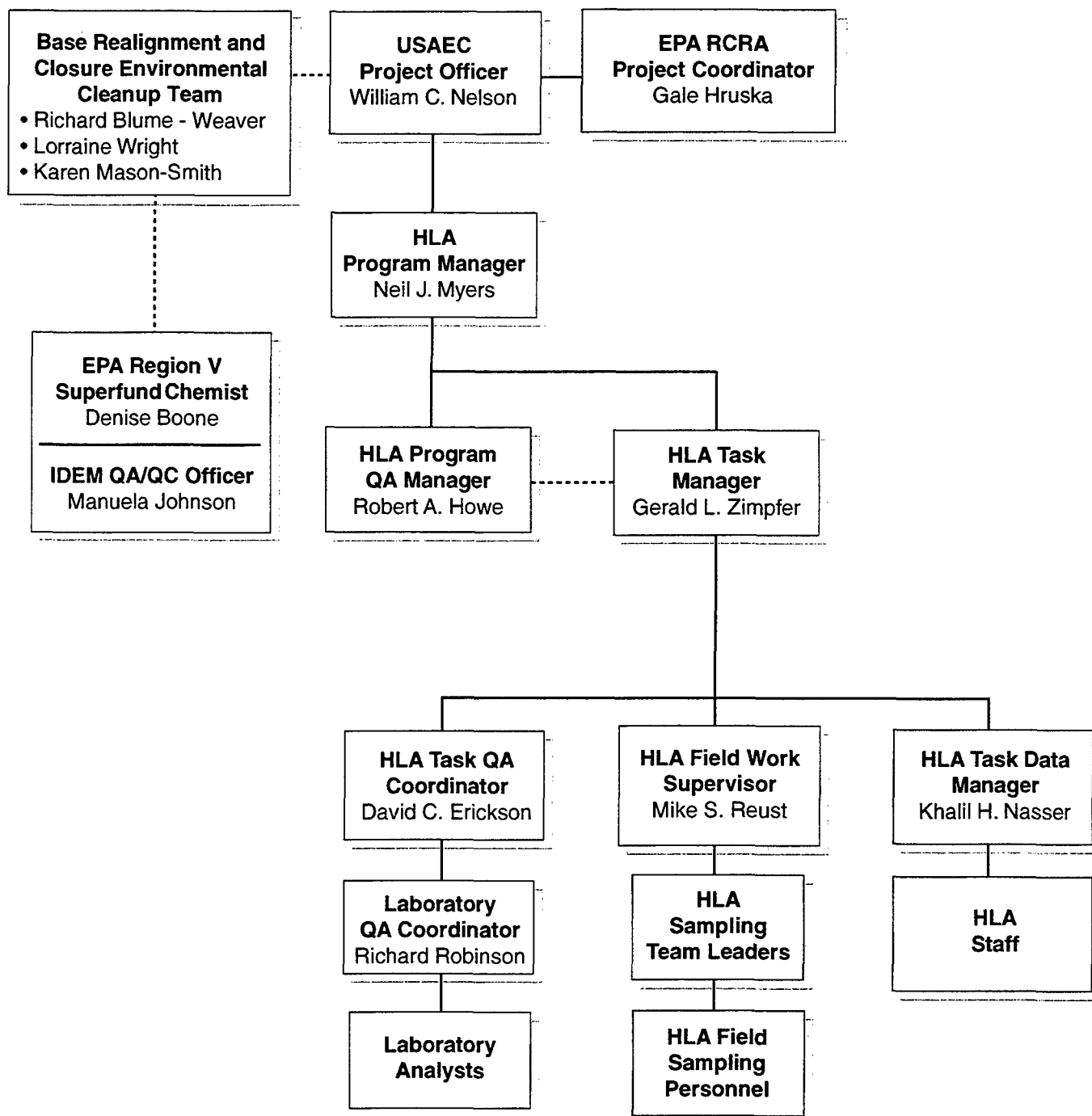
- Review and approve QAPjP.
- Review and evaluate analytical field and laboratory procedures.

HLA Program Manager - Neil J. Myers

- Technically review program deliverables.
- Review schedules, work plans, costs, and performance.
- Allocate resources to meet contract obligations.

HLA Task Manager - Gerald L. Zimpfer, Ph.D.

- Define task objectives and develop a detailed work plan schedule.
- Establish task policy and procedures to address the specific needs of the task as a whole, as well as the objectives of each task.
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints.
- Orient all field leaders and support staff concerning the task's special considerations.
- Monitor and direct the field leaders.
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product.



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Harding Lawson Associates
Engineering and
Environmental Services



Prepared for:
U.S. Army Environmental Center
Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
Marion County, Indiana

Figure 2.1
Quality Assurance Organizational Structure

Project Organization and Responsibility

- Review the work performed on each task to ensure its quality, responsiveness, and timeliness.
- Review and analyze overall task performance with respect to planned requirements and authorizations.
- Approve all reports (deliverables) before their submission to the Army.
- Ultimately be responsible for the preparation and quality of interim and final reports.
- Represent the task team at meetings and public hearings.

2.3 Quality Assurance Responsibilities

HLA Analytical Program Quality Assurance Manager - Robert A. Howe

- Coordinate TEPS analytical laboratory work.
- Prepare and review analytical data and data validation reports.
- Ensure that QA/QC procedures for program activities are conducted in a manner consistent with USAEC QA guidance and program QAPjP objectives.
- Provide recommendations concerning program QA objectives.
- Recommend corrective action procedures to maintain program QA objectives.
- Evaluate data deliverables for compliance with task-specific QAPjP requirements.
- Coordinate the implementation of laboratory and field audits.
- Review field and laboratory audit reports and assist in implementing corrective action identified by the field or laboratory audits.
- Provide technical guidance to the HLA Task Manager.

HLA Task Quality Assurance Coordinator - David C. Erickson

- Implement task QA requirements and coordinate field and laboratory data validation.
- Conduct field and laboratory audits to ensure that task QA program requirements are implemented.
- Coordinate with task management and Laboratory Quality Assurance Coordinators (LQACs) during task activities to ensure that QA requirements are being met.
- Ensure that corrective action is implemented when out-of-control situations are identified.
- Request any needed analytical reference materials from USAEC.
- Distribute task plans to key laboratory personnel.
- Perform announced and unannounced audits during sample collection.

Project Organization and Responsibility

- Check chain-of-custody records and lot designation forms for correctness and accuracy.
- Review analytical procedures and results to evaluate the analytical QC parameters of reported analytical results.
- Maintain an awareness of laboratory sample load to prevent a sample overload that could result in missed holding times and invalid analytical results.

HLA Task Data Manager - Khalil Nasser

- Convert electronic data to a format compatible with the Installation Restoration Data Management Information System (IRDMIS).
- Oversee entry of hardcopy data into the IRDMIS format.
- Oversee data checks to eliminate mistakes in electronic data files.
- Prepare electronic data files for transfer to USAEC.
- Transfer electronic data files to USAEC.

2.4 Laboratory Responsibilities

ESE Laboratory Quality Assurance Coordinator - Richard Robinson

- Act as liaison with the HLA Task QA Coordinator.
- Monitor laboratory workloads and ensure availability of resources.
- Review task chain-of-custody procedures to ensure that they match task requirements as stipulated in the associated planning documents.
- Oversee task laboratory activities to ensure the implementation of RFI analytical program requirements as specified in this QAPjP.
- Maintain a system to check the quality of laboratory materials, such as reagents and chemical supplies.
- Document and communicate with the HLA QA Coordinator regarding any unusual analytical results or situations that may require corrective action.

2.5 Field Responsibilities

HLA Field Supervisor - Mike S. Reust

- Oversee the implementation of task-required field QA/QC activities.
- Review field measurements for accuracy and precision.
- Review field logbooks for completeness and accuracy.

Project Organization and Responsibility

- Recommend corrective action procedures to maintain sample integrity.
- Coordinate field and sampling activities with the HLA Task Manager.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide results that are legally defensible in a court of law. Specific QA procedures for sampling, chain of custody, laboratory instrument calibration, laboratory analysis, data reporting, QC, laboratory audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPjP. The purpose of this section is to address the specific objectives for precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters. The definition and uses of PARCC parameters are also described in this section.

3.1 Level of Quality Control Effort

Rinse blank, trip blank, method blank, duplicate, standard reference material (SRM), and matrix spike (MS) samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Rinse and trip blanks consisting of distilled or deionized water will be submitted to the analytical laboratory to provide the means to assess the quality of the data resulting from the field sampling program. Rinse blank samples are analyzed to check for procedural contamination at the site that may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage.

Duplicate samples are analyzed to check for sampling and analytical reproducibility. MSs provide information about the effect of the sample matrix on the digestion and measurement methodology. All MSs for organic analyses and SW-846 analyses are performed in duplicate and are referred to as matrix spike/matrix spike duplicate (MS/MSD) samples. For target analyte list (TAL) metals analyses, an MS and an unspiked laboratory duplicate are analyzed rather than MS/MSD samples.

Quality Assurance Objectives for Measurement Data

The general level of the QC effort will be 1 rinse blank per day per matrix and sample equipment type. Field duplicates will be collected at the rate of 1 per every 10 or fewer investigative samples. One VOC trip blank consisting of distilled deionized ultra-pure water will be included along with each shipment of VOC analysis samples.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/ designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil). The number of field duplicate and field QC blank samples to be collected are described in Section 8.2 of this QAPjP. Sampling procedures are specified in Appendix A of the TSP.

The level of QA/QC adhered to by the laboratory will be equivalent to that specified under the Contract Laboratory Program (CLP) for Routine Analytical Services (RAS). The QC criteria for metals analyses (total and dissolved) in water (surface and ground) will conform to the protocols stipulated in EPA CLP Statement of Work (SOW) ILC01.0 for Low Concentration Water for Inorganic Analysis, and the QC criteria for metals analysis in soil will conform to the protocols stipulated in EPA CLP SOW ILM03.0 for Multi-media, Multi-concentration Inorganic Analysis. The QC criteria for organic analyses in water, including VOCs, SVOCs, and pesticides/PCBs, will be as stipulated in EPA CLP SOW OLC01.0 for Low Concentration Water for Organics Analysis. The QC criteria for organic analyses in soil for VOCs, SVOCs, and pesticides/PCBs will be as stipulated in EPA CLP SOW OLM03.1 for Multi-media, Multi-concentration Organic Analysis. The QC performance criteria for herbicides analyses in water, soil, and TPH analyses in soil will conform to the protocols of SW-846, Method 8150, and Method 8015 modified under an EPA CLP Special Analytical Services (SAS) designation. The QC performance criteria for dioxins/furans analyses in water and soil will be performed as stipulated in the SW-846, Method 8290 under an EPA CLP SAS designation. The QC performance criteria for TOC analyses of groundwater will conform to protocols of SW-846

Quality Assurance Objectives for Measurement Data

Method 9060 under an EPA CLP SAS designation. The QC performance criteria for cation exchange capacity analyses of soil will conform to the protocols of SW-846 Method 9080. For the measurements of pH, premeasurement calibrations and postmeasurement verifications will be performed using two standard reference solutions that will closely bracket the sample pH. This procedure will be performed for each sample tested. For field conductivity measurements, daily calibration of the instrument will be performed using standard solutions of known conductivity, in similar concentration ranges as the investigative sample.

3.2 Accuracy, Precision, and Sensitivity of Analysis

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

The accuracy and precision requirements for RAS parameters VOCs, SVOCs, and pesticides/PCBs are specified in the current SOW/OLC01.0 or OLM03.1 for organics, and accuracy and precision requirements for RAS metals are specified in the current SOW/ILC01.0 or ILM03.0 for inorganics, respectively. The accuracy and precision QC criteria for herbicide, dioxin/furan, TPH, TOC, and CEC analyses are specified in SW-846 Methods 8150, 8290 modified Method 8015, 9060, 9080, respectively. The analytical method sensitivities required for these analyses will be the Contract-Required Quantitation/Detection Limits (CRQLs or CRDLs) Practical Quantitation Limits (PQLs) or Method Detection Limits (MDLs) and are shown in Tables 3.1 through 3.7. Also shown, for comparison purposes, are the respective drinking water Maximum Contaminant Levels. A summary of the quality control criteria, including those for accuracy and precision for the respective analytical methods are summarized in Appendix C. The accuracy, precision, and sensitivity requirements for herbicides, dioxins/furans, TPH, and CEC are specified in the laboratory's standard operating procedures (SOPs) contained in Appendix A of this QAPjP. Method detection limits will be validated for each method before Phase II RFI samples are analyzed. Method detection limits will be assessed following the procedures described in 40 Code of Federal Regulations (CFR) Part 136, Appendix B "Definition and Procedure for the Determination of the Method Detection Limit." The SOPs for the

field equipment to measure pH, conductivity, and temperature are outlined in Appendix A of the TSP. Accuracy and precision requirements for field screening analyses are presented in Appendix C.

3.3 Completeness, Representativeness, and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that Environmental Science & Engineering, Inc. (ESE), will provide data meeting QC acceptance criteria for 90 percent or more for all samples tested using the RAS and methods listed in Section 3.1 of this QAPjP. Following completion of the analytical testing, the percent completeness will be calculated by the equation presented in Section 12.1 of this QAPjP.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that depends on the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to (1) results of the Phase I RFI and (2) Army and agency comments made during preparation of the Phase II TSP.

The rationale for investigative sampling at each investigation site is discussed in detail in the TSP. Representativeness will be satisfied by assuring that the TSP is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicate samples.

Comparability is an expression of the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned

Table 3.1: Target Compound List Analytes and Contract-Required Quantitation Limits for Volatile Organic Compounds

Compound	CAS Number	Quantitation Limits ^a			Maximum Contaminant Level ^d (µg/l)
		Water ^b (µg/l)	Low Soil ^c (µg/kg)	Medium Soil ^c (µg/kg)	
1. Chloromethane	74-87-3	1	10	1,200	---
2. Bromomethane	74-83-9	1	10	1,200	---
3. Vinyl chloride	75-01-4	1	10	1,200	2
4. Chloroethane	75-00-3	1	10	1,200	---
5. Methylene chloride	75-09-2	2	10	1,200	5
6. Acetone	67-64-1	5	10	1,200	---
7. Carbon disulfide	75-15-0	1	10	1,200	---
8. 1,1-Dichloroethene	75-35-4	1	10	1,200	7
9. 1,1-Dichloroethane	75-34-3	1	10	1,200	---
10. cis-1,2-Dichloroethene	156-59-4	1	---	---	70
11. trans-1,2-Dichloroethene	156-60-5	1	---	---	100
12. 1,2-Dichloroethene(total)	540-59-0	---	10	1,200	---
13. Chloroform	67-66-3	1	10	1,200	100
14. 1,2-Dichloroethane	107-06-2	1	10	1,200	5
15. 2-Butanone	78-93-3	5	10	1,200	---
16. Bromochloromethane	74-97-5	1	---	---	---
17. 1,1,1-Trichloroethane	71-55-6	1	10	1,200	200
18. Carbon tetrachloride	56-23-5	1	10	1,200	5
19. Bromodichloromethane	75-27-4	1	10	1,200	100
20. 1,2-Dichloropropane	78-87-5	1	10	1,200	5
21. cis-1,3-Dichloropropene	10061-01-5	1	10	1,200	---
22. Trichloroethene	79-01-6	1	10	1,200	5
23. Dibromochloromethane	124-48-1	1	10	1,200	100
24. 1,1,2-Trichloroethane	79-00-5	1	10	1,200	5
25. Benzene	71-43-2	1	10	1,200	5
26. trans-1,3-Dichloropropene	10061-02-6	1	10	1,200	---
27. Bromoform	75-25-2	1	10	1,200	100
28. 4-Methyl-2-pentanone	108-10-1	5	10	1,200	---
29. 2-Hexanone	591-78-6	5	10	1,200	---
30. Tetrachloroethene	127-18-4	1	10	1,200	5
31. Toluene	108-88-3	1	10	1,200	1,000
32. 1,1,2,2-Tetrachloroethane	79-34-5	1	10	1,200	---
33. Chlorobenzene	108-90-7	1	10	1,200	100
34. 1,2-Dibromoethane	106-93-4	1	---	---	0.05
35. Ethyl benzene	100-41-4	1	10	1,200	700
36. Styrene	100-42-5	1	10	1,200	100
37. Xylenes (total)	1330-20-7	1	10	1,200	10,000
38. 1,3-Dichlorobenzene	541-73-1	1	---	---	600
39. 1,4-Dichlorobenzene	106-46-7	1	---	---	75
40. 1,2-Dichlorobenzene	95-50-1	1	---	---	600
41. 1,2-Dibromo-3-chloropropane	96-12-8	1	---	---	0.2

--- Required Quantitation Limit not specified for this medium; laboratory will report lowest achievable quantitation limit
CAS Chemical Abstracts Service
µg/kg Micrograms per kilogram
µg/l Micrograms per liter

- Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil, calculated on dry-weight basis as required by the contract, will be higher.
- Environmental Protection Agency Statement of Work OLCO1.0 analytical method.
- Environmental Protection Agency Statement of Work OLMO3.1 analytical method.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Table 3.2: Target Compound List Analytes and Contract-Required Quantitation Limits for Semivolatile Organic Compounds

Compound	CAS Number	Quantitation Limits ^a			Maximum Contaminant Level ^e (µg/l)
		Water ^b (µg/l)	Low Soil ^c (µg/kg)	Medium Soil ^c (µg/kg)	
42. Phenol	108-95-2	5	330	10,000	---
43. bis(2-Chloroethyl) ether	111-44-4	5	330	10,000	---
44. 2-Chlorophenol	95-57-8	5	330	10,000	---
45. 1,3-Dichlorobenzene	541-73-1	---	330	10,000	600
46. 1,4-Dichlorobenzene	106-46-7	---	330	10,000	75
47. 1,2-Dichlorobenzene	95-50-1	---	330	10,000	600
48. 2-Methylphenol	95-48-7	5	330	10,000	---
49. 2,2'-oxybis-(1-Chloropropane) ^d	108-60-1	5	330	10,000	---
50. 4-Methylphenol	106-44-5	5	330	10,000	---
51. N-Nitroso-di-n-dipropylamine	621-64-7	5	330	10,000	---
52. Hexachloroethane	67-72-1	5	330	10,000	---
53. Nitrobenzene	98-95-3	5	330	10,000	---
54. Isophorone	78-59-1	5	330	10,000	---
55. 2-Nitrophenol	88-75-5	5	330	10,000	---
56. 2,4-Dimethylphenol	105-67-9	5	330	10,000	---
57. bis(2-Chloroethoxy) methane	111-91-1	5	330	10,000	---
58. 2,4-Dichlorophenol	120-83-2	5	330	10,000	---
59. 1,2,4-Trichlorobenzene	120-82-1	5	330	10,000	70
60. Naphthalene	91-20-3	5	330	10,000	---
61. 4-Chloroaniline	106-47-8	5	330	10,000	---
62. Hexachlorobutadiene	87-68-3	5	330	10,000	---
63. 4-Chloro-3-methylphenol	59-50-7	5	330	10,000	---
64. 2-Methylnaphthalene	91-57-6	5	330	10,000	---
65. Hexachlorocyclopentadiene	77-47-4	5	330	10,000	50
66. 2,4,6-Trichlorophenol	88-06-2	5	330	10,000	---
67. 2,4,5-Trichlorophenol	95-95-4	20	830	25,000	---
68. 2-Chloronaphthalene	91-58-7	5	330	10,000	---
69. 2-Nitroaniline	88-74-4	20	830	25,000	---
70. Dimethylphthalate	131-11-3	5	330	10,000	---
71. Acenaphthylene	208-96-8	5	330	10,000	---
72. 2,6-Dinitrotoluene	606-20-2	5	330	10,000	---
73. 3-Nitroaniline	99-09-2	20	830	25,000	---
74. Acenaphthene	83-32-9	5	330	10,000	---
75. 2,4-Dinitrophenol	51-28-5	20	830	25,000	---
76. 4-Nitrophenol	100-02-7	20	830	25,000	---
77. Dibenzofuran	132-64-9	5	330	10,000	---
78. 2,4-Dinitrotoluene	121-14-2	5	330	10,000	---
79. Diethylphthalate	84-66-2	5	330	10,000	---
80. 4-Chlorophenyl-phenyl ether	7005-72-3	5	330	10,000	---
81. Fluorene	86-73-7	5	330	10,000	---
82. 4-Nitroaniline	100-01-6	20	830	25,000	---
83. 4,6-Dinitro-2-methylphenol	534-52-1	20	830	25,000	---
84. N-Nitrosodiphenylamine	86-30-6	5	330	10,000	---
85. 4-Bromophenyl-phenyl ether	101-55-3	5	330	10,000	---
86. Hexachlorobenzene	118-74-1	5	330	10,000	1
87. Pentachlorophenol	87-86-5	20	830	25,000	1
88. Phenanthrene	85-01-8	5	330	10,000	---
89. Anthracene	120-12-7	5	330	10,000	---
90. Carbazole	86-74-8	---	330	10,000	---
91. Di-n-butylphthalate	86-74-2	5	330	10,000	---
92. Fluoranthene	206-44-0	5	330	10,000	---

Table 3.2 (continued)

Compound	CAS Number	Quantitation Limits ^a			Maximum Contaminant Level ^a (µg/l)
		Water ^b (µg/l)	Low Soil ^c (µg/kg)	Medium Soil ^c (µg/kg)	
93. Pyrene	129-00-0	5	330	10,000	---
94. Butylbenzylphthalate	85-68-7	5	330	10,000	---
95. 3,3-Dichlorobenzidine	91-94-1	5	330	10,000	---
96. Benzo(a)anthracene	56-55-3	5	330	10,000	---
97. Chrysene	210-81-9	5	330	10,000	---
98. bis(2-Ethylhexyl)phthalate	117-81-7	5	330	10,000	---
99. Di-n-octylphthalate	117-84-0	5	330	10,000	---
100. Benzo(b)fluoranthene	205-99-2	5	330	10,000	---
101. Benzo(k)fluoranthene	207-08-9	5	330	10,000	---
102. Benzo(a)pyrene	50-32-8	5	330	10,000	0.2
103. Indeno(1,2,3-cd)pyrene	193-39-5	5	330	10,000	---
104. Dibenzo(a,h)anthracene	53-70-3	5	330	10,000	---
105. Benzo(g,h,i)perylene	191-24-2	5	330	10,000	---

--- Required Quantitation Limit not specified for this medium; laboratory will report lowest achievable quantitation limit
CAS Chemical Abstracts Service
µg/kg Micrograms per kilogram
µg/l Micrograms per liter

- Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil, calculated on dry-weight basis as required by the contract, will be higher.
- Previously known by the name of bis(2-Chloroisopropyl) ether.
- Environmental Protection Agency Statement of Work OLCO1.0 analytical method.
- Environmental Protection Agency Statement of Work OLMO3.0 analytical method.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Table 3.3: Target Compound List Analytes and Contract-Required Quantitation Limits for Chlorinated Pesticides and Polychlorinated Biphenyls

Compound	CAS Number	Quantitation Limits ^a		Maximum Contaminant Level ^d ($\mu\text{g/l}$)
		Water ^b ($\mu\text{g/l}$)	Soil ^c ($\mu\text{g/kg}$)	
106. alpha-BHC	319-84-6	0.01	1.7	---
107. beta-BHC	319-85-7	0.01	1.7	---
108. delta-BHC	319-86-8	0.01	1.7	---
109. gamma-BHC (lindane)	58-89-9	0.01	1.7	0.2
110. Heptachlor	76-44-8	0.01	1.7	0.4
111. Aldrin	309-00-2	0.01	1.7	---
112. Heptachlor epoxide	1024-57-3	0.01	1.7	0.2
113. Endosulfan I	959-98-8	0.01	1.7	---
114. Dieldrin	60-57-1	0.02	3.3	---
115. 4,4-DDE	72-55-9	0.02	3.3	---
116. Endrin	72-20-8	0.02	3.3	2
117. Endosulfan II	33213-65-9	0.02	3.3	---
118. 4,4-DDD	72-54-8	0.02	3.3	---
119. Endosulfan sulfate	1031-07-8	0.02	3.3	---
120. 4,4-DDT	50-29-3	0.02	3.3	---
121. Methoxychlor	72-43-5	0.10	17.0	40
122. Endrin ketone	53494-70-5	0.02	3.3	---
123. Endrin aldehyde	7421-36-3	0.02	3.3	---
124. alpha-Chlordane	5103-71-9	0.01	1.7	---
125. gamma-Chlordane	5103-74-2	0.01	1.7	---
126. Toxaphene	8001-35-2	1.0	170.0	3
127. Aroclor-1016	12674-11-2	0.20	33.0	---
128. Aroclor-1221	11104-28-2	0.20	67.0	---
129. Aroclor-1232	11141-16-5	0.40	33.0	---
130. Aroclor-1242	53469-21-9	0.20	33.0	---
131. Aroclor-1248	12672-29-6	0.20	33.0	---
132. Aroclor-1254	11097-69-1	0.20	33.0	---
133. Aroclor-1260	11096-82-5	0.20	33.0	---

$\mu\text{g/l}$ Micrograms per liter
 $\mu\text{g/kg}$ Micrograms per kilogram

- Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil, calculated on dry-weight basis as required by the contract, will be higher. There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides.
- Environmental Protection Agency Statement of Work OLCO1.0 analytical method.
- Environmental Protection Agency Statement of Work OLMO3.1 analytical method.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Table 3.4: Target Compound List Analytes and Practical Quantitation Limits for Chlorinated Herbicides

Compound	CAS Number	Practical Quantitation Limits ^a		Maximum Contaminant Level ^c (µg/l)
		Water ^b (µg/l)	Soil ^b (mg/kg)	
2,4-D (2,4-Dichlorophenoxy acetic acid)	94-75-7	0.126	0.020	70
2,4-DB(4-[2,4-Dichlorophenoxy]butyric acid)	94-82-6	0.126	0.020	---
2,4,5-T ([2,4,5-Trichlorophenoxy] acetic acid)	93-72-1	0.126	0.020	---
2,4,5-TP (Silvex)	93-76-5	0.126	0.020	50
Dalapon	75-99-0	0.126	0.020	200
Dicamba	1918-00-9	0.126	0.020	---
Dichloroprop	120-36-5	0.126	0.020	---
Dinoseb	88-85-7	0.126	0.020	7
MCPA ([4-Chloro-2-methylphenoxy] acetic acid)	94-74-6	3.0	0.40	---
MCPP ([±]-2-[4-Chloro-2-methyl phenoxy] propanoic acid)	93-65-2	3.0	0.40	---

CAS Chemical Abstracts Service
mg/kg Milligrams per kilogram
µg/l Micrograms per liter

- Sample practical quantitation limits (PQLs) are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable. For nonaqueous samples, the PQL is on a wet-weight basis.
- Environmental Protection Agency SW-846 8150 analytical method.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Table 3.5: Inorganic Target Analyte List Analytes and Contract-Required Detection Limits for Metals and Cyanide

Analyte	CAS Number	Detection Limit ^a		Maximum Contaminant Level ^d ($\mu\text{g/l}$)
		Water ^b ($\mu\text{g/l}$)	Soil ^c (mg/kg)	
Aluminum	7429-90-5	100	40	---
Antimony	7440-36-0	5	12	6
Arsenic	7440-38-2	2	2	50
Barium	7440-39-3	20	40	2,000
Beryllium	7440-41-7	1	1	4
Cadmium	7440-43-9	1	1	5
Calcium	7440-47-3	500	1,000	---
Chromium	7440-70-2	10	2	100
Cobalt	7440-48-4	10	10	---
Copper	7440-50-8	10	5	1,300
Iron	7439-89-6	100	20	---
Lead	7439-92-1	2	0.6	15
Magnesium	7439-95-4	500	1,000	---
Manganese	7439-96-5	10	3	---
Mercury	7439-97-6	0.2	0.04	2
Nickel	7440-02-0	20	8	100
Potassium	7440-02-7	750	1,000	---
Selenium	7882-49-2	3	1	50
Silver	7440-22-4	10	2	---
Sodium	7440-23-5	500	1,000	---
Thallium	7440-28-0	10	2	2
Vanadium	7440-62-2	10	10	---
Zinc	7440-66-6	20	4	---
Cyanide	---	10	2	200

CAS Chemical Abstracts Service
mg/kg Milligrams per kilogram
 $\mu\text{g/l}$ Micrograms per liter

- Detection limits listed for soil are based on wet weight. The detection limits calculated by the laboratory for soil, calculated on a dry-weight basis as required by the contract, will be higher.
- Environmental Protection Agency Statement of Work ILCO1.0 analytical method.
- Environmental Protection Agency Statement of Work ILMO3.0 analytical method.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Table 3.6: Target Analytes and Practical Quantitation Limits for Additional Parameters and Total Petroleum Hydrocarbons

Parameters	Practical Quantitation Limits ^a	
	Water ($\mu\text{g/l}$)	Soil (mg/kg)
Additional Parameters		
Total organic carbon ^b	1,000	NA
Cation exchange capacity ^c	NA	TBD
Total Petroleum Hydrocarbons^d		
Gasoline	400	8
Diesel	400	8
Motor Oil	400	8

mg/kg Milligrams per kilogram
 $\mu\text{g/l}$ Micrograms per liter
 NA Information not available
 TBD To be determined

- Sample practical quantitation limits (PQLs) are highly matrix- and method detection limit (MDL) - dependent. The PQLs listed herein are provided for guidance and may not always be achievable. For nonaqueous samples, the PQL is on a wet-weight basis.
- Environmental Protection Agency SW-846 9060 analytical method.
- Environmental Protection Agency SW-846 9080 analytical method.
- Environmental Protection Agency SW-846 Modified 8015 analytical method.

Table 3.7: Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans Analyses by SW-846 Method 8290

Compound	Detection Limits ^a		Maximum Contaminant Level ^c (µg/l)
	Water (ppq)	Soil (ppt)	
Polychlorinated dibenzo-p-dioxins ^b			
Total heptachloro-dibenzo-p-dioxin (HpCDD)	25 to 50	2.5 to 5.0	---
Total hexachloro-dibenzo-p-dioxin (HxCDD)	25 to 50	2.5 to 5.0	---
Total octachloro-dibenzo-p-dioxin (OCDD)	50 to 100	5.0 to 10	---
Total pentachloro-dibenzo-p-dioxin (PeCDD)	25 to 50	2.5 to 5.0	---
Total tetrachloro-dibenzo-p-dioxin (TCDD)	5 to 10	0.5 to 1.0	---
Polychlorinated dibenzofurans ^b			
Total heptachloro-dibenzo-p-furan (HpCDF)	25 to 50	2.5 to 5.0	---
Total hexachloro-dibenzo-p-furan (HxCDF)	25 to 50	2.5 to 5.0	---
Total octachloro-dibenzo-p-furan (OCDF)	50 to 100	5.0 to 10	---
Total pentachloro-dibenzo-p-furan (PeCDF)	25 to 50	2.5 to 5.0	---
Total tetrachloro-dibenzo-p-furan (TCDF)	5 to 10	0.5 to 1.0	---
Specific isomers			
1,2,3,4,6,7,8-HpCDD	25 to 50	2.5 to 5.0	---
1,2,3,4,6,7,8-HpCDF	25 to 50	2.5 to 5.0	---
1,2,3,4,7,8-HxCDD	25 to 50	2.5 to 5.0	---
1,2,3,4,7,8-HxCDF	25 to 50	2.5 to 5.0	---
1,2,3,4,7,8,9-HpCDF	25 to 50	2.5 to 5.0	---
1,2,3,6,7,8-HxCDD	25 to 50	2.5 to 5.0	---
1,2,3,6,7,8-HxCDF	25 to 50	2.5 to 5.0	---
1,2,3,7,8-PeCDD	25 to 50	2.5 to 5.0	---
1,2,3,7,8-PeCDF	25 to 50	2.5 to 5.0	---
1,2,3,7,8,9-HxCDD	25 to 50	2.5 to 5.0	---
1,2,3,7,8,9-HxCDF	25 to 50	2.5 to 5.0	---
2,3,4,6,7,8-HxCDF	25 to 50	2.5 to 5.0	---
2,3,4,7,8-PeCDF	25 to 50	2.5 to 5.0	---
2,3,7,8-TCDD	5 to 10	0.5 to 1.0	3 x 10 ⁻⁵
2,3,7,8-TCDF	5 to 10	0.5 to 1.0	---

--- Not available

ppq Parts per quadrillion (picograms per liter)

ppt Parts per trillion (nanograms per kilogram)

- The detection limit ranges are estimates. Actual detection limits for each congener will be sample specific and will be reported by the laboratory.
- Environmental Protection Agency Analytical Method SW-846 8290.
- Maximum contaminant levels for drinking water, Office of Water, U.S. Environmental Protection Agency, shown for comparison purposes.

Quality Assurance Objectives for Measurement Data

analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives.

4.0 SAMPLING PROCEDURES

Investigative samples will be collected during the RFI from various FBH locations and media. The media to be sampled and analyzed include soil and groundwater. The number and location of samples to be collected and the rationale for collecting them is provided in the TSP. In addition, field operations affecting sample collection also are addressed in greater detail in the TSP. The soil and groundwater samples collected as part of the RFI will be analyzed for the presence of VOCs, SVOCs, pesticides/PCBs, dioxins, herbicides, and metals. Section 5.0 of the TSP provides a summary of samples and analytical parameters for the Phase II RFI. Sampling procedures are described in Appendix A of the TSP and are summarized below.

All activities performed and observations made during sampling will be documented as part of a sample management program. Sampling activities will be documented to verify that sample integrity is maintained during sample collection, transportation, and storage before analysis. Documentation in this sample management program will be used to provide a record of procedures used in sample collection and analysis. Sample collection procedures and documentation are discussed in the following sections.

4.1 Field Sampling by Matrix

Subsurface soil, surface soil and groundwater, and surface soil for PCB screening and for VOC screening samples will be collected during the RFI. Sampling procedures and documentation are described below.

4.1.1 Subsurface-Soil Sample Collection

Subsurface-soil samples collected from borings will be retained in 6-inch-long by 2.5-inch-diameter stainless-steel liners. Soil samples will be collected directly in these liners by driving an 18-inch-long split-barrel sampler equipped with three liners into the ground at the specified sample depth. Soil samples will be collected for chemical analysis from the intervals specified in the TSP. Subsurface-soil sampling will be accomplished using a truck-mounted drilling rig equipped with

Sampling Procedures

hollow-stem augers. Subsurface-soil sample collection procedures are described in greater detail in Appendix A of the TSP.

4.1.2 Surface-Soil Sample Collection

Surface-soil samples will be collected for analysis using a stainless-steel trowel. Surface-soil samples will be collected from the 0.0- to 0.5-foot surface interval of each discrete sampling location.

Composite surface-soil samples will not be collected during the RFI. Surface-soil sample collection procedures are described in greater detail in Appendix A of the TSP.

4.1.3 Groundwater Sample Collection

Groundwater samples will be collected from new and existing monitoring wells. Newly installed groundwater monitoring wells will be sampled no sooner than seven days after well development.

All wells will be purged in accordance with procedures provided in Appendix A of the TSP.

Groundwater monitoring well samples will be collected using either a bailer or pump. Groundwater collected from monitoring wells will include analyses for total and dissolved metals. Groundwater samples collected for dissolved metals will be filtered in the field. Groundwater monitoring well installation, development, and sampling procedures are described in greater detail in Appendix A of the TSP.

4.1.4 Polychlorinated Biphenyl Screening Soil Sample Collection

Surface-soil samples for PCB screening will be collected at designated locations from the 0.0- to 0.5-foot depth interval using a stainless-steel trowel. Additional soil sampling information for the PCB screening is presented in Appendix A of the TSP.

4.1.5 Volatile Organic Compounds Field Screening Soil Sample Collection

Selected surface-soil samples will be screened for VOCs in the field. Results of the VOC screening will be used to decide whether subsequent laboratory VOC analyses are warranted. The screening test for VOCs will consist of an ambient temperature headspace analysis. A 16-ounce glass jar will be half filled with freshly sampled soil, sealed with a clean sheet of aluminum foil, agitated for at

least 30 seconds, and allowed to reach the ambient temperature (75 degrees Fahrenheit [$^{\circ}$ F]). A description of the screening procedure is provided in Section 7.2.2.

4.1.6 Excavated Soil Sample Collection

Subsurface-soil samples will be collected from trenches excavated during the investigation of subsurface geophysical anomalies. Soil will be removed from trenches using a decontaminated backhoe bucket. Soil samples for laboratory analysis will be removed from the backhoe bucket and placed in an appropriate sample container using a decontaminated stainless-steel trowel. The sampling locations will be documented as described in the TSP.

4.2 Field Quality Control Sample Collection/Preparation Procedures

Field QC, including rinse blanks, field blanks, trip blanks, field duplicates, matrix spike, and MS/MSD samples, is collected at the frequency specified in Section 3.1 of this QAPjP. Rinse blanks will be prepared in the field from water used during the decontamination of sampling equipment. Field blanks will be prepared in the field from potable and deionized or distilled water used during the sampling event. Rinse and field blanks will be analyzed for those target analytes evaluated in the associated investigative samples.

Trip blanks pertain to aqueous VOC samples only. They are prepared by the laboratory using analyte-free water and are sent into the field with the empty sample bottles. They are kept with the investigative samples throughout the sampling event, packaged for shipment to the laboratory, and analyzed along with the investigative samples. Trip blanks should be included in each shipping container that contains aqueous samples for VOC analysis. At no time after preparation are trip blanks to be opened before they are received by the laboratory for analysis.

Field duplicates are independent samples collected so they are equally representative of the parameter(s) of interest at a given point in space and time. When collected, processed, and analyzed by the same organization, these samples provide intralaboratory precision information for the entire

measurement system, including sample acquisition, matrix homogeneity, handling, shipping, storage, preparation, and analysis. They can also be used to estimate the overall precision of a data collection activity.

MS/MSDs and MSs are collected from the matrix of the associated investigative samples. Sample collection, containers, and preservation for both the MS and MSD samples are the same as for the associated investigative samples.

4.3 Sample Containers, Preservatives, and Volume Requirements

This section includes summaries of sample containers, preservatives, and volume requirements by medium of samples collected for chemical or physical analyses. All samples will be collected in containers that are free of all project target compounds. The laboratory will provide sample bottles and vials, purchased from commercial sources, that will be certified by the supplier as analyte-free for project target compounds. Sample container preparation procedures are described in Table 4.1. The sample container supply company will provide a certified analysis for each sample container lot. The reuse of bottles is expressly prohibited. The cleaning process for liners is discussed in Appendix A of the TSP.

4.3.1 Subsurface-Soil Samples

Each subsurface-soil sample will be collected in one 6-inch by 2.5-inch stainless-steel liner. The liners will be labeled with the information described in Section 5.1 of this QAPjP before samples are collected. The stainless-steel liners will be sealed with Teflon®-lined caps and inserted into large resealable plastic bags immediately after sampling to prevent the caps from coming off or moisture from entering the liners during shipment. Shipping soil-bore samples to the laboratory in stainless-steel liners requires less handling of the samples in the field and reduces the chances of sample contamination and the loss of sample integrity.

Table 4.1: Sample Container Cleaning Procedures Within the Laboratory

Analysis/Parameter	Container Type	Matrix	Cleaning Protocol*
Organic extractables including GC and GC/MS	Glass jar with Teflon®-lined cap	Water	A
	Glass jar with Teflon®-lined cap	Soil/sediment	A
Organic purgeables including GC and GC/MS analyses	Glass septum vial with Teflon®-lined cup	Water	B
	Wide-mouth glass jar with Teflon®-lined cap	Soil	B
Metals	Linear polyethylene cubitainer with polyethylene cap	Water	C
	Glass jar with Teflon®-lined cap (or new plastic)	Soil/sediment	A
Inorganics including total cyanide	Linear polyethylene cubitainer with polyethylene cap	Water	D
	Glass jar with Teflon®-lined cap (or new plastic)	Soil	A

Cleaning Protocol				Specifications
A	B	C	D	
X	X	X		Wash with hot tap water using laboratory-grade, interference-free, nonphosphate detergent.
X	X	X		Rinse 3 times with tap water.
X		X		Rinse with 1:1 nitric acid (reagent-grade nitric acid diluted with ASTM Type 1 deionized water).
X	X	X		Rinse 3 times with ASTM Type 1 deionized water.
X				Rinse with pesticide-grade methylene chloride using 20 ml per 64-oz. bottle, 10 ml per 32- or 16-oz bottle, or 5 ml per 8- or 4-oz bottle. Methylene chloride is used as organics rinse.
X		X		Oven dry, using a forced-air oven, at 105° to 125°C for 1 hour.
		X		Invert and air-dry in contaminant-free environment.
X		X	X	The containers are sealed with caps containing Teflon® liners or Teflon®-backed septa that had been cleaned the same way as the containers, packed in cartons, and stored until needed.
			X	No cleaning required; use new cubitainers only.

Cleaning protocols A, B, and C are applied by commercial supplier. Cleaning protocol D is applied by ESE.

Source: Environmental Science and Engineering, Inc.

GC/HPLC Gas chromatography/high performance liquid chromatography

GC/MS Gas chromatography/mass spectrometry

Glass Amber for all organic water analyses

No chemical preservation of the subsurface soil samples will be required; however, samples will be maintained at $4 \text{ degrees } \pm 2 \text{ Celsius } (^{\circ}\text{C})$ by placing the samples under ice in coolers and shipping via overnight delivery to the analytical laboratory. Subsurface soil samples will be collected in accordance with specifications presented in Table 4.2.

4.3.2 Surface and Excavated Soil Samples

Each surface or excavated soil sample will be collected in 125-ml or 250-ml, wide-mouth amber glass bottles so that each bottle is completely filled. Soil will be removed from the lip of the bottles to ensure an air-tight seal when they are capped. The bottles will be labeled with the information described in Section 5.1 before the samples are collected.

No chemical preservation of the soil samples will be required; however, samples will be maintained at $4^{\circ} \pm 2 \text{ C}$ by placing the samples under ice in coolers and shipping via overnight delivery to the analytical laboratory. Surface or excavated soil samples will be collected in accordance with the specifications provided in Table 4.3.

4.3.3 Groundwater Samples

Sample bottles used to collect groundwater samples will be labeled with the information described in Section 5.1 before the samples are collected. Sample bottles for analytes other than VOCs will be triple-rinsed with the sample medium before being filled; preservatives will then be added. Sample bottles for VOCs will not be triple-rinsed before being filled, and preservatives will be added before the bottles are filled. The groundwater samples will be collected in accordance with the specifications listed in Table 4.4. VOC vials will be capped with Teflon[®]-lined septum caps so that no air bubbles are trapped in the vials. Samples will be maintained at $4^{\circ} \pm 2 \text{ C}$ by placing the samples under ice in coolers and shipping via overnight delivery to the analytical laboratory.

Table 4.2: Sample Container, Preservation, and Holding Times for Subsurface Soil Sample Analyses

Analysis	Sample Container*	Preservation	Holding Time
VOCs	Stainless-steel liner	4°C	Analyze within 10 days of sample receipt
SVOCs	Stainless-steel liner	4°C	Extract within 10 days of sample receipt Analyze within 40 days of extraction
Pesticides/PCBs	Stainless-steel liner	4°C	Extract within 10 days of sample receipt Analyze within 40 days of extraction
Herbicides	Stainless-steel liner	4°C	Extract within 14 days of sample collection Analyze within 40 days of extraction
Dioxins/furans	Stainless-steel liner	4°C	Extract within 30 days of sample collection Analyze within 45 days of extraction
Metals	Stainless-steel liner	4°C	Analyze within 180 days of sample receipt (except mercury, 26 days after sample receipt)
Cyanide	Stainless-steel liner	4°C	Analyze within 12 days of sample receipt
TPH	Stainless-steel liner	4°C	Extract within 14 days of sample collection Analyze within 40 days of extraction
Cation Exchange Capacity	Stainless-steel liner	4°C	Not specified
Total Organic Carbon	Stainless-steel liner	4°C	Analyze within 28 days of sample collection

°C Degrees Celsius
 PCB Polychlorinated biphenyl
 SVOC Semivolatile organic compound
 VOC Volatile organic compound

* One stainless-steel liner will contain sufficient sample volume to perform the described analyses.

Table 4.3: Sample Container, Preservation, and Holding Times for Surface and Excavated Soil Sample Analyses

Analysis	Sample Container	Preservation	Holding Time
VOCs	125-ml amber glass	4° C	Analyze within 10 days of sample receipt
SVOCs	250-ml amber glass	4° C	Extract within 10 days of sample receipt Analyze within 40 days of extraction
Pesticides/PCBs	250-ml amber glass	4° C	Extract within 10 days of sample receipt Analyze within 40 days of extraction
Herbicides	250-ml amber glass	4° C	Extract within 14 days of sample collection Analyze within 40 days of extraction
Dioxins/furans	250-ml amber glass	4° C	Extract within 30 days of sample collection Analyze within 45 days of extraction
Metals	250-ml amber glass	4° C	Analyze within 180 days of sample receipt (except mercury, 26 days after sample receipt)
Cyanide	250-ml amber glass	4° C	Analyze within 12 days of sample receipt
TPH	250-ml amber glass	4° C	Extract within 14 days of sample collection Analyze within 40 days of extraction
Cation Exchange Capacity	250-ml amber glass	4° C	Not specified
Total Organic Carbon	250-ml amber glass	4° C	Analyze within 28 days of sample collection

°C Degrees Celsius
ml Milliliter
PCB Polychlorinated biphenyl
SVOC Semivolatile organic compound
TPH Total petroleum hydrocarbons
VOC Volatile organic compound

Table 4.4: Sample Container, Preservation, and Holding Times for Groundwater Sample Analyses

Analysis	Sample Container	Preservation	Holding Time
VOCs	Three 40-ml glass	Add HCl (pH <2); store at 4° C (add Na ₂ S ₂ O ₃ , if needed, for residual chlorine)	Analyze within 10 days of sample receipt
SVOCs	Two 1-liter amber glass	Store at 4° C	Extract within 5 days of sample receipt Analyze within 40 days of extraction
Pesticides/PCBs	Two 1-liter amber glass	Store at 4° C	Extract within 5 days of sample receipt Analyze within 40 days of extraction
Herbicides	One 1-liter amber glass	Store at 4° C	Extract within 7 days of sample collection Analyze within 40 days of extraction
Dioxins and furans	Two 1-liter amber glass	Store at 4° C	Extract within 30 days of sample collection Analyze within 45 days of extraction
Metals	One 1-liter plastic	Add 0.5 ml HNO ₃ (pH <2); store at 4° C	Analyze within 180 days of sample receipt (except mercury, 26 days after sample receipt)
Cyanide	One 1-liter plastic	Add NaOH (pH >12); store at 4° C	Analyze within 12 days of sample receipt
TPH	Three 40-ml glass	Add HCl (pH <2); store at 4° C	Extract sample within 7 days of sample collection Analyze within 40 days of extraction
Specific conductivity, Eh	1-liter plastic		Measure immediately
Total organic carbon	1-liter amber glass	Add H ₂ SO ₄ (pH<2); store at 4° C	Analyze within 28 days of sample collection

> Greater than
< Less than
°C Degrees Celsius
H₂SO₄ Sulfuric acid
HCl Hydrochloric acid
HNO₃ Nitric acid
ml Milliliter
Na₂S₂O₃ Sodium thiosulfate
NaOH Sodium hydroxide
PCB Polychlorinated biphenyl
SVOC Semivolatile organic compound
TPH Total petroleum hydrocarbons
VOC Volatile organic compound

5.0 SAMPLE CUSTODY

It is EPA policy to follow the EPA Region V sample custody, or chain-of-custody protocols as described in "NEIC Policies and Procedures," EPA-330/9-78-DDI-R, revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if:

- The item is in actual possession of a person.
- The item is in the view of the person after being in actual possession of the person.
- The item was in actual physical possession but is locked up to prevent tampering.
- The item is in a designated and identified secure area.

5.1 Sample Collection Custody Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the chain of custody intact. The protocol for specific sample numbering and other sample designations are included in the TSP, Appendix B.

5.1.1 Initiation of Chain-of-Custody Field Procedures

The field sample custody procedures discussed here are an integral part of the record-keeping procedures described below. The samples must be adequately identified for sample custody to be tracked. Sample labels, sample tags, and chain-of-custody forms will bear the same type of information for all media. Figures 5.1, 5.2, 5.3, 5.4, and 5.5 are examples of the groundwater sampling form, chain-of-custody, sample label, label for sample shipment and sample of airbill form, respectively, that will be used to label and identify samples collected during the RFI. The chain-of-custody form is designed to accommodate the following:

- Computer preprinting

Sample Custody

- Multiple containers per sample
- Preservation of samples

Field chain-of-custody procedures are summarized below:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to the HLA Field Supervisor or properly dispatched. As few people as possible should handle the samples.
2. All sample bottles will be tagged with sample numbers and location. The sample (tag number) number and sample label will be affixed.
3. Sample tags will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint would not function in freezing weather.
4. The HLA Field Supervisor will review all field activities to assess whether proper custody procedures were followed during the fieldwork and decide whether additional samples are required.

5.1.2 Chain-of-Custody Forms and Sample Labels

All sample bottles will be labeled, and each sample label and chain-of-custody form will bear, at a minimum, the following information:

1. Sample location and depth
2. Media type
3. Site identification (ID), which is keyed into a location with an alphanumeric code or by a descriptive title
4. Project code: an assigned HLA project number
5. Date: a six-digit number indicating the day, month, and year of collection
6. Time: a four-digit number indicating the 24-hour clock time of collection
7. Depth from which the sample was obtained, if applicable
8. Sampling technique
9. Name and signature of personnel responsible for sample collection
10. Specific chemical or physical analysis to be performed
11. Project name (chain of custody only)

Log Book # _____ Page _____ of _____									
Well No.:	Purge Equipment <input type="checkbox"/> Bennett Pump (Teflon Tubing) <input type="checkbox"/> ISCO Pump (Teflon Tubing) <input type="checkbox"/> Standard Pump (PVC Tubing) <input type="checkbox"/> Grundfos Pump (Neoprene Tubing) <input type="checkbox"/> Stainless Bailer	pH Meter: <input type="checkbox"/> Beckman phi 21 <input type="checkbox"/> Omega pH-65A <input type="checkbox"/> Orion SA250 <input type="checkbox"/> Other _____ SERIAL NO. _____	Date: _____ HLA Project No.: _____ Location: _____						
Casing Diameter in.	O.D. LENGTH 1.65" <input type="checkbox"/> 2 ft. <input type="checkbox"/> 1.85" <input type="checkbox"/> 3 ft. <input type="checkbox"/> 3.75" <input type="checkbox"/> 4 ft. <input type="checkbox"/> _____ ft. _____ SERIAL NO. _____	Conductivity Meter: <input type="checkbox"/> YSI Model 33 <input type="checkbox"/> Other _____ SERIAL NO. _____	Meter Calibration _____ Time _____ pH 7.00= _____ at _____ °C						
Casing Stickup ft.			pH 10.00= _____ at _____ °C						
Total Well Depth ft.			Conductance Standard: _____ μmhos/cm at 25° C Time _____ Measured Value: _____ μmhos/cm at _____ °C Calibrated Conductivity = Measured Conductance + (0.02) (measured conductance) (25° C - Actual Temp): _____ Time _____ _____ μmhos/cm at 25° C						
Static Water Level ft.	Sample Equipment <input type="checkbox"/> Same as Purge <input type="checkbox"/> Bennett Pump (Teflon Tubing) <input type="checkbox"/> ISCO Pump (Teflon Tubing) <input type="checkbox"/> Standard Pump (PVC Tubing) <input type="checkbox"/> Stainless Bailer	Dissolved Oxygen Meter: <input type="checkbox"/> YSI Model 51B SERIAL NO. _____	Dissolved Oxygen _____ mg/l at _____ °C						
Saturated Thickness ft.			Titration Results (Acid Concentration: <input type="checkbox"/> 0.16, <input type="checkbox"/> 1.6) pH 8.3 5.1 4.8 4.5						
Casing Volume gal.			#Clicks _____ Color _____						
Screened Interval ft.	O.D. LENGTH 1.65" <input type="checkbox"/> 2 ft. <input type="checkbox"/> 1.85" <input type="checkbox"/> 3 ft. <input type="checkbox"/> 3.75" <input type="checkbox"/> 4 ft. <input type="checkbox"/> _____ ft. _____ SERIAL NO. _____	Filtration Equipment: <input type="checkbox"/> Geotech Peristaltic Pump <input type="checkbox"/> Geotech 0.45 micron filter	Eh Measurement _____ Time: _____ Millivolt Reading _____						
			Water Level Meter: Solinst _____ HLA# _____						
Time	Number of Casing Volumes	Gallons Removed	°C	E.C. μmhos/cm	pH	Dissolved O ₂ mg/Liter	Pump Rate gpm	Approx. Pump Depth ft.	Visual Description
Analysis Requested Volatile Aromatics Volatile Organohalogenes Organosulfur Compounds Organochlorine Pesticides Phosphonates Hydrocarbons Anions Nitrate/Nitrite (0.5 ml H ₂ SO ₄) Arsenic (0.5 ml HNO ₃) Mercury (0.5 ml HNO ₃) ICP Metals (0.5 ml HNO ₃) Acid Extractables Cyanide (1 ml NaOH) GC/MS Volatiles GC/MS SV/Acid Extractables Nit/Phos Pesticides						Health and Safety Officer Comments: _____ Health and Safety Officer Signature: _____ Condition of Well, Remarks: _____ Sampler Signature: _____		Sample Depth (cm): _____ Protective Level: D C B	

Harding Lawson Associates
Engineering and
Environmental Services



Prepared for:
U.S. Army Environmental Center
Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
Marion County, Indiana

Figure 5.1
Groundwater Sampling Form


[illegible]

Revised 05/15/96

FILE

Figure 5.2

Example Chain-of-Custody Form

Site ID:	 Harding Lawson Associates 707 Seventeenth Street Denver, Colorado 80202 303/292-5365		
Site Type:			
Sample Tech:			
Depth (cm):			
Data:	<div>Analysis</div> <div>Container</div> <div>Preservative</div>		
Time:	Remarks:		Tag No:
	Sampler Signature:		

Revised 05/15/96

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 Engineering and
 Environmental Services



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 U.S. Army Environmental Center
 Aberdeen Proving Ground, Maryland

 Fort Benjamin Harrison
 Marion County, Indiana

Figure 5.3
 Example Sample Label

OTHER REGULATED SUBSTANCES

Harding Lawson Associates
 707 Seventeenth Street
 Suite 2400
 Denver, Colorado 80202
 (303) 292-5365

ID8027

Revised 05/15/96

Harding Lawson Associates
 Engineering and
 Environmental Services



Prepared for:
 U.S. Army Environmental Center
 Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
 Marion County, Indiana

Figure 5.4

Identification Label for Sample Shipment

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Figure 5.5
Sample Courier Airbill Form

Revised 5/15/96



12. Bill of lading number (chain of custody only)
13. Laboratory ID (chain of custody only)
14. Unique sample tag number
15. Preservation technique used and whether sample has been filtered

5.1.3 Field Logbooks and Documentation

Field logbooks enable field personnel to record data collection activities as they are performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory. Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to individual field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will bear the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- Project end date

Entries into the logbooks will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site as well as field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a

Sample Custody

single strike mark, initialed, and dated. A comment as to why the entry was crossed out should also be made. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes, when available, compass and distance measurements, shall be recorded. The number of photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sample procedures documented in Appendix A of the TSP and summarized in Section 4.0. Information to be recorded during sample collection activities includes:

- Equipment used to collect samples
- Sample collection times
- Sample collection depth
- Description of sample
- Sample volume and number of sample containers
- Quality control samples collected

Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

5.1.4 Transfer of Custody and Shipment Procedures

1. Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory (if present), to a permanent laboratory, or to and from a secure storage area.
2. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals for shipment to the laboratory. The preferred procedure includes the use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

Sample Custody

3. Whenever samples are collocated with a source or a government agency, a separate Sample Receipt is prepared for those samples and marked to indicate with whom the samples are being collocated. The person relinquishing the samples to the facility or agency should request the representative signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.
4. All shipments will be accompanied by the chain-of-custody form identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to HLA.
5. If samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the chain-of-custody form as long as it is sealed inside the sample cooler and the custody seal remains intact.

5.2 Laboratory Chain-of-Custody Procedures

Samples collected in the field will be labeled and tracked according to matrix. Water samples will be tracked separately from soil samples. Procedures to be used by each subcontractor laboratory must be consistent with standard chain-of-custody practices previously used for USAEC programs.

Standard tracking procedures or SOPs will be provided by the subcontractor laboratories to the contractor and must be approved by the HLA Task QA Coordinator and USAEC before analyses begin. Automated sample control programs may be substituted for manual chain-of-custody procedures but must provide the same information and high degree of reliability as a manual system.

A laboratory sample tracking or control system will address the following elements:

1. A sample receipt officer or custodian
2. An individual responsible for samples
3. Analyses request information
4. Field sample number
5. Location of sample storage at all times
6. Date sample was received or retrieved by analyst
7. Present condition or step of analysis being performed on a sample
8. Date analysis was completed
9. Signature of analyst

Sample Custody

10. Laboratory ID number
11. Bottle tag number
12. Report date
13. Special instructions for analysis

The laboratory will appoint a LQAC to ensure the listed information is collected and maintained throughout a project. The LQAC will be responsible for sample integrity while the sample is in the custody of the laboratory. The LQAC will maintain a permanent record of all identifying sample tags, data sheets, and laboratory records. Any problems with project samples will be communicated by the LQAC to the HLA Task QA Coordinator immediately after receipt and inspection of samples by the sample custodian. Samples will be logged into a separate logbook at the laboratory that will contain the following information:

1. Unique laboratory sample ID number
2. USAEC project name
3. Date and time of sample receipt
4. Analysis requested
5. Volume of sample received
6. Number of containers received per sample
7. Type and condition of sample container
8. Observations concerning sample condition, including broken containers, leakage, and temperature
9. Location where samples will be stored
10. Bill of lading number
11. Signature of sample custodian

The sample custodian and analyst will be responsible for samples in their custody. The sample custodian will be responsible for inspecting samples for breakage after receipt at the laboratory. The sample custodian will transfer custody of the samples to the appropriate section supervisor so lot

assignments and analysis request forms can be issued. Samples will be distributed into a secure storage facility, and the chain-of-custody forms will be signed by the appropriate section supervisor. Samples will be logged in or out of this storage facility for analyses or proper disposal. Any sample splitting or extraction will be entered onto the chain-of-custody form or laboratory extraction bench logbook. Extraction or processing logbooks will be stored with all other required raw data deliverables in a secure storage facility until the final transfer of data to USAEC is performed.

5.2.1 Laboratory Sample Lot and Sample Analysis Numbers

Analytical results will be submitted to USAEC for processing through the non-THAMA approved method (NTAMs) database system. For compatibility with the NTAMs database system, analytical results produced by the laboratory will be assigned a lot designation and a sample analysis number. The lot designation, consisting of four letters in a unique sequence (e.g., QARB), will be used to identify a group of samples for reporting analytical results. The lot designation will refer to a group of 20 or fewer field samples of the same matrix, submitted for a common analysis (e.g., VOCs), and received over a period of up to 5 calendar days. Analytical results for all samples in the lot are reported concurrently. A lot is defined by one of the following, whichever occurs first:

- Each 20 field samples within a sample medium for a single analytical method or extraction type
- Each 5-day calendar period during which field samples are received

5.3 Final Evidence Files and Custody Procedures

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this QAPjP. HLA will transfer project documents to USAEC at the conclusion of the RFI. USAEC is the custodian of the evidence file and will maintain the contents of evidence files for the RFI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews in a secured, limited access area and under custody of the USAEC facility manager.

The final evidence file will include, at a minimum, the following:

- Field logbooks
- Field data and data deliverables
- Photographs
- Drawings
- Soil boring logs
- Laboratory data deliverables
- Data validation reports
- Data assessment reports
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, bill of lading, etc.)

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy of the instruments and measuring equipment that are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled, periodic basis.

6.1 Field Instruments and Equipment

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Equipment to be used during the field sampling activities will be examined to assess that it is in satisfactory operating condition. This examination includes checking the manufacturer's operating manual and instructions for each instrument to ensure that maintenance requirements are observed. Field notes from previous sampling activities will be reviewed so that the notation on any previous equipment problem is not overlooked and to ensure that necessary repairs to equipment have been performed. Instruments meeting these requirements will be given a serialized number and made available for project use. Instruments and equipment not meeting satisfactory operating conditions are labeled as such and are withheld from project use until they are modified or repaired to meet project requirements. A list of calibration standards, including source, traceability, and verification of purity must be included in the field instrument logbook.

Calibration of field instruments is governed by specific SOPs for the applicable field analysis method, and such procedures take precedence over the following general discussion.

Each item of equipment used in field activities will be calibrated at a frequency specified by the appropriate SOP or by the owner/operator manual provided by the manufacturer. Equipment calibration is recorded by HLA field personnel in a bound field instrument logbook and contains at a minimum the following information:

Calibration Procedures and Frequency

- Equipment ID
- Control number
- Calibration schedule and frequency
- Equipment specifications
- Specification verification (where applicable)
- Equipment necessary to accomplish calibration
- Procedure for calibration
- Corrective action
- Data pertaining to the calibration procedures
- Date of calibration
- Initials of analyst performing calibration
- Adjustments made to the equipment before and after calibration
- Record of equipment failure or inability to meet specifications

General calibration requirements consistent with the manufacturers' owner/operator manuals for field equipment to be used for RFI field activities are discussed separately below. Field instruments will include water-level meter, pH meter, specific conductivity meter, thermometer, PCB field screening equipment, and organic vapor analyzer.

6.1.1 Water-level Measurements

The sounder will be checked against a steel surveyor's tape before use. The graduated steel tape will have the manufacturer-supplied temperature correction applied if field conditions warrant. The pressure transducer is calibrated at the factory, calibrated in house with water columns before aquifer tests, and checked weekly in the field against steel tape or against a sounder during use.

6.1.2 pH Measurements

The digital pH meter (Beckman Model 021 or equivalent) will be calibrated daily with two standard buffer solutions before field measurements. The range of the buffer solutions will be not more than

Calibration Procedures and Frequency

three or more pH units apart and will bracket the expected pH of the sample being measured.

Calibration procedures and frequency will be recorded in the field logbook along with the lot number of the buffer solutions.

General procedures for calibrating pH meters are as follows:

- Ensure that the temperature of the sample and buffer are the same.
- Connect pH electrode to pH meter and turn on the pH meter.
 - Adjust the temperature setting if temperature compensation is not automated on the basis of the buffer temperature, then place the electrode in the first buffer solution.
 - Adjust the calibration knob to display the correct value after the reading has been stabilized, remove the pH electrode from the buffer, rinse with distilled water, and pat dry.
- Repeat this procedure for the second buffer solution.
- Place the pH electrode in the sample and record the pH measurement displayed.
- Remove the pH electrode from the sample, rinse with distilled water, and pat dry.
 - Place the pH electrode in the first buffer solution to verify the calibration. Continue for each sample measurement as listed above, bracketing each sample reading with acceptable buffer verifications.
 - Recalibrate the pH meter every time it is turned off and turned back on, or if calibration verifications were not met.

The calibrations performed, standards used, and sample pH values measured will be recorded in the field logbook. New batteries will be purchased and kept with the meters to facilitate immediate replacement in the field as necessary.

6.1.3 Eh Measurement

Eh measurements of groundwater will be read directly in millivolts using an Orion Model 250 meter equipped with an Orion Redox probe (Model #96-78BN). Calibration of the meter for Eh millivolt measurement is not required. Eh measurements are readily affected by contact of the groundwater sample with the atmosphere. Consequently, Eh should be measured in undisturbed groundwater still contained within the well, or measured in groundwater pumped through a flow cell. The Orion

Calibration Procedures and Frequency

Model 250 meter may, when equipped with a pH probe, be used to also measure pH, following the general procedures listed above.

6.1.4 Specific Conductance

The conductivity cells of the specific conductivity meter (YSI Model SSB or equivalent) will be cleaned and checked daily against known conductivity standards before use. In the field, the instrument will be checked daily with National Institute of Standards and Technology (NIST) (or other approved sources) traceable reference standards. The calibration procedure follows:

- Place the probe in conductivity calibration standard solution.
- Set the temperature knob to the temperature of the standard solution.
- Turn to the appropriate scale and set the instrument for the calibration standard value.
- Rinse the conductivity electrode with distilled water and pat dry.
 - Measure the conductivity of distilled water, ensuring the temperature is set correctly for the temperature of the sample to be measured.
 - Remove the conductivity electrode and pat dry.
 - Measure field samples as above; after every 10 samples, confirm calibration with the measurement of the calibration standard solution.
 - If the scale of the instrument has to be changed due to the nature of the sample, the above calibration will be made for the newly chosen measuring scale and the appropriate standard will be measured before sample testing continues.

Sample readings and calibrations will be recorded in the field logbook.

6.1.5 Water Temperature

Temperature meters will be used to measure water temperatures. The temperature meters will be rechecked in the field before and after each use according to manufacturer's instructions.

Temperature meters will be checked biannually for calibration by immersing the temperature meter probe in a water bath of known temperature until equilibrium is reached. The reference thermometer used for the water bath calibration will be traceable to NIST calibration thermometers.

Calibration Procedures and Frequency

All temperature meters will be calibrated weekly with a mercury thermometer and calibrations will be recorded in the field logbook.

6.1.6 Polychlorinated Biphenyl Screening Instrument Calibration

PCB screening of surface-soil samples will be conducted by HLA personnel using a PCB field test kit. The field test procedure will be performed according to the PCB field test kit manufacturer's instructions. A summary of the PCB soil screening method is presented in Appendix A of the TSP. The designated PCB field test kit, an EnSys PCB RIS[®] Soil Test System, conforms to proposed EPA Method 4020 for immunoassay-based field screening for PCBs in soil. The method has a minimum detection level of 0.4 milligrams per kilogram (mg/kg) for PCB. The test system gives equal response at this level with Aroclors 1254 and 1260. Aroclors 1016, 1232, 1242, and 1248 are measured with minimum detection levels of 4, 4, 2, and 1 mg/kg, respectively. Because this test is specific for PCBs, no interferences are expected due to other chlorinated compounds that may be present at the site. The concentration of PCBs in the soil sample is quantified by the test as a colored end product. Color production is inversely proportional to the concentration of PCBs in the samples. The intensity of color is measured using a spectrophotometer. The spectrophotometer will be operated following manufacturer's instructions. The concentration of PCBs in the soil samples will be assessed using a two-point concentration curve of color intensity (optical density) and concentration of PCBs. Calibration will be verified every 10 samples.

6.1.7 Organic Vapor Analysis

The portable gas analyzers currently identified as being available for onsite use during field operations are the HNU Model PI-101 photoionization analyzers and the Foxboro Model 128 organic vapor analyzer equipped with a flame ionization detector. Equivalent instruments may also be used during the investigation. External standard calibration procedures specified in the factory supplied instruction manual will be followed. These procedures include calibrating the instrument with an appropriate calibration gas (e.g., isobutylene) in the concentration range expected to be used. The calibration gas will be used at ambient temperature and pressure. The instrument calibration will be

checked daily by using the internal calibration mechanism. Procedures for the calibration and operation of the organic vapor analyzers are provided in Appendix F of the Final Site Safety and Health Plan for the Phase II RFI (HLA, 1996b).

6.2 Laboratory Instruments

Laboratory equipment will be calibrated on the basis of method-specific procedures (Table 7.1). Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QA/QC activities. These records will be filed where the work is performed and will be subject to a QA audit. For instruments, the laboratory will maintain a factory-trained repair staff with in-house spare parts or will maintain service contracts with equipment vendors. The calibration records will be maintained as follows:

- If possible, each instrument will have a calibration record permanently affixed to it with an assigned record number.
- A label will be affixed to each instrument showing description, manufacturer, model numbers, date of last calibration, calibrator's signature, and due date of next calibration. Reports and compensation or correction figures will be maintained with the instrument.
- Written step-wise calibration procedures will be available for each measurement instrument.
- Any instrument that is not calibrated to the manufacturer's original specification will display a warning tag to alert the analyst that the instrument has only a limited calibration.

Before samples are analyzed on an instrument, chemical calibration standards of each target analyte must be analyzed to establish that the instrument is functioning properly with the desired sensitivity. As many analytes as possible are combined in the chemical calibration standards to provide (1) economy of effort for standards analyses, and (2) adequate evaluation of instrument performance, response, and sensitivity for multi-analyte analyses, as may be expected for samples.

Chemical instrument calibration will be accomplished using calibration standards prepared by mixing the species to be analyzed in the solvent that is introduced into the instrument, as dictated by the analytical method. The concentrations of the chemical calibration standards will be chosen to bracket the allowable linear reporting range of the method.

Calibration Procedures and Frequency

Data from the chemical calibration standards shall be plotted, as necessary, with the instrument response indicated on the ordinate and the concentration indicated on the abscissa. When microprocessors are used to establish calibration curves, the data must, nevertheless, be plotted. If, after plotting, the curve is shown to be linear with acceptable variance, the microprocessor may be used to determine analyte concentrations in samples. Methods and formulae for quantification will be as specified in the standardized methods. For organic gas chromatograph analyses, chemical instrument calibration curves will not be used to determine the linearity of the calibration. Rather, the analyst will use chemical calibration standards analyses to establish instrument responses versus concentration relationships, with early warning of instrument variances provided by the statistical distribution.

Data from the chemical instrument calibrations will be recorded on the appropriate forms and maintained with the lot data package. Alternatively, if a laboratorywide computerized data management system is available, calibration data may be generated electronically and output on forms or charts. In either case, documentation will be included with the lot data package to demonstrate the validity of the chemical instrument calibration.

6.2.1 Organic Analyses

This section describes procedures for maintaining laboratory instrumentation dedicated to the analysis of samples for organic compounds.

6.2.1.1 Gas Chromatography/Mass Spectrometry Tuning

Prior to calibration, the instrument(s) used for gas chromatography/mass spectrometry (GC/MS) analyses are tuned by analysis of bromofluorobenzene (BFB) for VOCs and decafluorotriphenyl phosphine (DFTPP) for SVOCs. Once the tuning criteria for these reference compounds are met, the instrument should be initially calibrated following criteria specific to each analytical method. The instrument tune will be verified each 12 hours of operation.

The criteria for acceptability will be as specified in the respective EPA analytical methods.

6.2.1.2 Initial Calibration

Initial calibration procedures will be used whenever the following occurs or as specified by the specific analytical method:

- The MDL is determined.
- The instrument is started up (other than daily startup and shutdown).
- The instrument is used to analyze analytes different from those for which the instrument was previously calibrated.
- The instrument fails daily calibration.

Initial calibration will be as specified in the analytical method. If calibration requirements are not specified in the method, then contact HLA or the USAEC Chemistry Branch for guidance.

6.2.1.3 Daily Calibration

Calibration standards will be analyzed each day, prior to sample analysis, to verify that the instrument response has not changed from the previous calibration. Daily calibration will be performed in accordance with the requirements of the appropriate analytical method and must fall within the limit of acceptability stated within. If calibration requirements are not specified in the method, then contact HLA or the USAEC Chemistry Branch for guidance.

After the tuning criteria are met (GC/MS methods), the instrument is initially calibrated following procedures specific to the analytical method. The calibration standards will be EPA or NIST traceable.

6.2.1.4 Continuing Calibrations

Continuing calibration will be performed in accordance with the analytical methods listed in Section 3.1 of this QAPjP and will include the following:

Calibration Procedures and Frequency

- **GC/MS VOCs** - an instrument blank and a continuing calibration standard will be analyzed directly after an acceptable instrument tune. The standard will meet the limits of acceptability as defined in EPA CLP SOW OLC01.0 (water) or EPA CLP SOW OLM03.1 (soil).
- **GC/MS SVOCs** - a continuing calibration standard will be analyzed directly after an acceptable instrument tune. The standard will meet the limits of acceptability as defined in EPA CLP SOW OLC01.0 (water) or EPA CLP/SOW OLM03.1 (soil).
- **Pesticides/PCBs or PCBs** - an instrument blank will be analyzed every 12 hours. In addition, every 12 hours, the laboratory will alternately analyze a performance evaluation mixture (PEM) and the individual standard mixtures A and B. All results for these standards will meet the limits of acceptability as defined in EPA CLP SOW OLC01.0 (water) or EPA CLP/SOW OLM03.1 (soil).
- **Herbicides** - a mid-level calibration standard will be analyzed every 10 samples. The standard will meet the limits of acceptability as defined in SW-846, Method 8150.
- **Dioxins/Furans** - Once the GC/MS system has been calibrated, the calibration must be verified for each 12-hour period of operation.

The continuing calibration consists of two parts: evaluation of the chromatographic resolution and verification of the RRF values to be used for quantitation. At the beginning of each 12-hour period, the chromatographic resolution is verified in the same fashion as in the initial calibration.

- **Total Petroleum Hydrocarbons** - A calibration standard will be analyzed after every 10 samples and at the end of the run. Analysis response must be within 15 percent of the same concentration standard analyzed during the initial calibration.

If a continuing calibration fails to meet the limits of acceptability, the laboratory will immediately take corrective action, including recalibration of the instrument and reanalyses of all samples analyzed since the last acceptable calibration.

6.2.2 Inorganic Analyses

This section describes procedures for maintaining laboratory instrumentation dedicated to the analysis of samples for inorganic compounds.

6.2.2.1 Initial Calibration

Initial calibration procedures will be used whenever the following occurs:

- The MDL is determined.
- The instrument is started up (other than daily startup and shutdown).

- The instrument is used to analyze analytes different from those for which the instrument was previously calibrated.
- The instrument fails daily calibration.

6.2.2.2 Daily Calibration

Calibration standards will be analyzed each day, prior to sample analysis, to verify that the instrument response has not changed from the previous calibration. Daily calibration will be performed in accordance with the requirements of the analytical method and must fall within the limits of acceptability as stated. If calibration requirements are not within those limits specified in the method, then contact HLA or the USAEC Chemistry Branch for guidance.

The graphite furnace atomic absorption (GFAA) spectrophotometer is calibrated using a minimum of three calibration standards prepared by dilution of certified stock solutions and an analysis blank.

The calibration standards bracket the concentration range of the samples. Calibrate at least daily or each time the instrument is set up. The ICP spectrophotometer is calibrated per instrument manufacturer's specifications. The cold vapor atomic absorption (CVAA) spectrophotometer is calibrated using a minimum of four standards and one analysis blank.

The instruments used for performing CEC and TOC analyses will be calibrated according to the specifications in the analytical methods from EPA SW-846 or EPA Standards Methods, as appropriate, and the respective laboratory SOPs. A minimum of three calibration standards and one analysis blank will be used to calibrate the instruments, with one calibration standard at the contract-required quantitation limit (CRQL), contract-required detection limit (CRDL), or Practical Quantitation Limit (PQL) as applicable for each analyte of interest. The calibration standards bracket the concentration range of the samples.

The linearity near the CRDL for the TAL metals will be verified with a standard prepared at a concentration of two times the quantitation limit (one times the quantitation limit for the metals

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analyzed by graphite furnace and mercury). This standard must be run at the beginning and at the end of each sample analysis run or a minimum of twice per eight-hour period. For mercury and the metals analyzed by graphite furnace, this standard is only analyzed at the beginning of the sample analysis run, but not before the Initial Calibration Verification. Corrective action is taken when QC limits for the initial calibration verification (ICV) and/or continuing calibration verification (CCV) are not met, which may include recalibrating the instrument and reanalyzing the previous 10 samples.

6.2.2.3 Initial Calibration Verification Standards

ICVs are prepared from different stock solution than the calibration or CCV standards and are EPA or NIST traceable.

ICV standards are required for inorganic analyses, and will be analyzed following each initial calibration. The ICV standard contains all analytes of interest for the method in question at a concentration near one to two times the CRQL. Examples of the limits of acceptability for different types of calibration check standards are described in the associated analytical methods.

Corrective action is taken when QC limits for ICV are not met, which may include recalibrating the instrument and reanalyzing all samples analyzed since the last acceptable calibration.

6.2.2.4 Continuing Calibration Verification Standards

Continuing calibration will be performed in accordance with the analytical methods listed in Section 3.1 of this QAPjP and will include the following:

- **Inorganics (metals and cyanide)** - a continuing calibration blank (CCB) and a CCV standard will be analyzed for every 10 samples or every two hours, whichever is more frequent. The standard will be near the midpoint of the Method Reporting Range (MRR) and will meet the limits of acceptability as specified EPA CLP SOW ILC01.0 (water) or EPA CLP SOW ILM03.0 (soil and sediment).
- **Inorganics** - a CCB and CCV standard will be analyzed for every ten samples or every two hours, whichever is more frequent. The standard will be near the midpoint of the MRR and will meet the limits of acceptability as defined in EPA SW-846 or EPA Standard Methods for the respective. The CCV results should meet a minimum requirement of ± 15 percent of the value for acceptance.

If a continuing calibration fails to meet the limits of acceptability, the laboratory will immediately take corrective action, including recalibration of the instrument and reanalyses of all samples analyzed since the last acceptable calibration.

6.3 Reference Materials

During chemical calibration and sample analyses, solutions containing known analytes at known concentrations must be prepared. These solutions are needed to generate method startup data, calibrate instruments, spike samples and standards with analytical surrogates and/or internal standards, prepare QC samples, and prepare Performance Evaluation samples, when specified. Three types of reference materials may be used to prepare standard solutions, as described in the following sections. Before initiating any laboratory studies, the laboratory must submit a request to the USAEC Project Officer or Contracting Officer's Representative (COR) for reference materials. The list should include all target analytes of interest on a specific project, surrogate compounds, and internal standards. The USAEC Project Officer or COR will forward the request to the USAEC Chemistry Branch. Samples of reference materials will be shipped to the laboratory from the repository. Only if reference materials are not available through USAEC should the subcontractor laboratory obtain the materials from an outside source. Reference materials for metals and nonmetallic inorganics may be maintained at room temperature in a locked storage area. All other reference materials must be stored in a locked refrigerator at or below 4°C. All reference materials shall be maintained under chain of custody. An SOP for the use, control, and inventory of reference materials will be prepared and stored by the subcontractor laboratory.

6.3.1 Standard Analytical Reference Materials

Whenever possible, chemical analyses conducted in support of the RFI will be based on standard analytical reference materials (SARMs). These materials are labeled as SARMs and carry a SARM identification number. These materials will either be NIST SARMs or will be traceable to NIST SARMs. Contractors are encouraged to use secondary standards that are referenced to SARMs and are periodically checked against SARMs. This check will be performed the first time the standard is

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used and at six-month intervals or when the standard is replaced, whichever comes first. The use of secondary standards is encouraged as a conservation method for the more costly SARMs.

6.3.2 Interim Reference Materials

Interim reference materials (IRMs) are available from two sources. Some of these materials are maintained and distributed by USAEC and should be used if SARMs are not available. Although IRMs are supplied through USAEC, they are not as rigorously characterized as SARMs. IRM characterization includes positive identification of the material and an estimate of purity. The SARM label on each bottle is modified by adding the word "Interim" and includes an ID number. These materials may be used as received from USAEC. Reference materials obtained from NIST do not require characterization by the laboratory.

6.3.3 Off-the-Shelf Materials

SARMs or IRMs may not be available for some target analytes. If materials are unavailable through USAEC, subcontractor laboratories will be instructed to purchase materials from an outside supplier. Before using any material, regardless of source, classified as "off-the-shelf," the laboratory must analyze the material to obtain a positive identification and estimate of purity. Where possible, characterization analyses for purity shall be conducted using at least two different methods. Off-the-shelf materials should be compared to NIST or EPA standard material whenever possible. Documentation for purity and identity characterization analyses shall be kept on file at the subcontractor laboratory. Possible techniques for characterizing the off-the-shelf materials include the following, as applicable:

- Infrared spectroscopy
- Melting point, decomposition point, or boiling point determinations
- Mass spectrometry
- Elemental analysis
- Gas chromatography (for purity)
- Liquid chromatography (for purity)

This list is not exhaustive, and all of the listed techniques need not be used. The subcontractor laboratory is responsible for providing positive identification and a purity estimate for each off-the-shelf material (including internal standards).

6.3.4 Analytical Records for Reference Materials

As previously indicated, SARMs and QC analytical reference materials must be tracked in a bound logbook. This record must include date of receipt, source, purity, label information, storage conditions, and expiration date. This logbook should also maintain a record of reference material performance. Similarly, reagents used in sample preparation must also be logged and their performance tracked. A standard preparation logbook will also be maintained for reference materials as well as each type of QC sample. This logbook will contain details concerning QC sample preparation and will include at a minimum the following types of information:

- Chemical Abstracts Service (CAS) number of each analyte
- Solvent used
- Solvent lot number
- Source of stock
- Concentration of stocks used in preparation
- Dilutions performed
- Final concentration of QC sample
- Initials of chemist preparing the solution
- Date of preparation and expiration date for QC sample

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

Soil and groundwater samples collected during field sampling activities for the RFI will be analyzed by Environmental Science & Engineering, Inc. (ESE), P.O. Box 1703, Gainesville, Florida, 32602-1703, Phone (904) 332-3318. Table 7.1 provides a summary of the analytical methods that will be used during the Phase II RFI.

Before an analytical method can be used for this project, the subcontractor laboratory must demonstrate the ability to perform the method for the specified analytes. The laboratory will determine a MDL for each analyte of interest using the procedures described in 40 CFR, Part 136, Appendix B (EPA, 1984). The MDL determination procedures are summarized as follows:

- The laboratory will prepare a standard matrix sample at one to five times the estimated MDL.
- A minimum of seven replicates of the sample will be processed through the entire method.
- The laboratory will calculate the standard deviation of results from the seven (or more) replicate samples.
- The MDL is equal to the standard deviation multiplied by the student's test value (e.g., 3.143 for seven replicates).

The MDL will be equal to or less than the respective EPA PQLs for EPA SW-846 methods or contract required detection or quantitation limits (CRDLs or CRQLs) for CLP methods. The laboratory must provide a summary of the results of the current MDL study for the target analytes. MDL studies over one year old or performed using instrumentation other than that proposed for use on this project must be repeated prior to the analysis of project samples.

7.1 Laboratory Analysis

Sample analysis shall be performed following the appropriate CLP RAS and SAS methods as explained in the following sections.

7.1.1 CLP RAS Laboratory Analysis

All samples for CLP TCL VOCs, SVOCs, pesticides/PCBs and CLP TAL metals will be analyzed according to analytical procedures set forth in the EPA CLP RAS SOW (OLC01.0 for water [surface and ground], and OLM03.1 for soil [surface and subsurface] and sediment) for organic analysis and RAS SOW (ILC01.0 for water [surface and ground], and ILM03.0 for soil [surface and subsurface]) for inorganic analysis.

A complete listing of project target compounds and laboratory reporting limits for each analyte group listed in Table 7.1 can be found in Section 3.0. Laboratory reporting limits are specified in the respective CLP SOW for organic analysis and inorganic analysis and in the laboratory SOPs found in Appendix A for herbicide, TPH, dioxin/furan, and inorganic parameters analyses. The respective SOWs for organic and inorganic analysis specify associated QC samples experimentally determined and are on file at the laboratory.

7.1.2 Special Analytical Services Laboratory Analysis

The general guidelines and procedures for SAS analyses of herbicides, dioxins/furans, TPH, TOC, and CEC parameters analyses are detailed in the sections that follow.

7.1.2.1 Herbicide Analysis

Samples for herbicides will be analyzed according to the analytical procedures set forth in the EPA SW-846 Method 8150 under an EPA CLP SAS designation. The laboratory SOP for the herbicide sample preparation and gas chromatography (GC) analysis are included in Appendix A of this QAPjP. The laboratory is following EPA SW-846, Method 8150 for preparation and analysis, and Method 8000 for general calibration and QC requirements. The following QC criteria will be met:

- Daily retention time windows will be established for both of the analytical GC columns used.
- The laboratory will provide method-specific MS and surrogate recovery limits, which are located in Appendix C.
- A continuing calibration verification check standard (CCS) will be analyzed after every 10 samples. QC samples like MS, LCS, or blanks count as a sample. If a CCS exceeds the

**Table 7.1: Proposed Analytical Methods for the Phase II
Fort Benjamin Harrison RFI**

Analytical Parameters	Proposed Analytical Method	
	Water	Soil
Volatile organic compounds	CLP SOW OLC01.0	CLP SOW OLM03.1
Semivolatile organic compounds	CLP SOW OLC01.0	CLP SOW OLM03.1
Pesticides	CLP SOW OLC01.0	CLP SOW OLM03.1
Polychlorinated biphenyls	CLP SOW OLC01.0	CLP SOW OLM03.1
Herbicides	SW-846, 8150 ^{a,b}	SW-846, 8150 ^{a,b}
Total metals	CLP SOW ILC01.0	CLP SOW ILM03.0
Dissolved metals	CLP SOW ILC01.0	CLP SOW ILM03.0
Cyanide	CLP SOW ILC01.0	CLP SOW ILM03.0
Dioxins/furans	SW-846, 8290 ^b	SW-846, 8290 ^b
Total petroleum hydrocarbons	SW-846 Modified 8015 ^{a,b}	SW-846 Modified 8015 ^{a,b}
Total organic carbon	SW-846 9060 ^a	SW-846 9060 ^a
Cation exchange capacity	---	SW 846 9080 ^a

CLP SOW Contract Laboratory Program Statement of Work

a. SW-846, 8000 will be used for general calibration and quality control.

b. USEPA 1994, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods SW-846.

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limit of ± 15 percent of the true value, the analyst has to take corrective action, which may include reanalyses of the previous 10 samples.

- Confirmation of a tentative identified analyte will occur on a secondary column. Any decision of the analyst to reject the tentative identification will be clearly documented in the data package; if necessary, enlargements of chromatograms will be submitted.

7.1.2.2 Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Analyses

Samples for dioxin/furan analyses will be analyzed according to the analytical procedures set forth in the EPA SW-846 Method 8290 under an EPA CLP SAS designation. The laboratory SOP for sample preparation and high resolution GC/MS analysis are included in A of this QAPjP. Method-specific QC criteria are presented in Appendix C. The following QC criteria will be met:

- A GC column performance check is required at the beginning of each 12-hour period during which samples are analyzed. Column performance check criteria specified in the method shall be met.
- A method blank run is required between a calibration run and the first sample run.
- The mass spectrometer performance shall be monitored by reviewing static resolution and mass drifts. Mass spectrometer performance must meet method-specific criteria.
- Performance check solutions will be analyzed at the beginning of each 12-hour period during which samples are run. If required method-specific criteria are not met, remedial action must be taken before any samples are analyzed.
- Duplicate sample analyses (percent recovery and concentrations of 2,3,4,8 substituted dioxin/furan congeners) should agree within 25 percent relative difference.
- Matrix spike/matrix spike duplicate sample analyses will be performed. Concentrations of 2, 3, 7, 8 substituted dioxin/furan congeners should agree within 20 percent relative percent difference (Appendix C).
- Internal standards analyses should be monitored and the percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards (Appendix C).

7.1.2.3 Total Petroleum Hydrocarbons Analysis

Samples for TPH will be analyzed according to the analytical procedures set forth in the EPA SW-846 Modified Method 8015 under an EPA CLP SAS designation. The laboratory SOP for the TPH sample preparation and GC analysis are included in Appendix A of this QAPjP. The laboratory is following

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EPA SW-846, Modified Method 8015 for preparation and analysis, and Method 8000 for general calibration and QC requirements. The following QC criteria will be met:

- Daily retention time windows will be established for both of the analytical GC columns used.
- The laboratory will provide method-specific MS recovery limits, which are located in Appendix C.
- A continuing calibration verification check standard (CCS) will be analyzed after every 10 samples. QC samples like MS, LCS, or blanks count as a sample. If a CCS exceeds the limit of ± 15 percent of the true value, the analyst has to take corrective action, which may include reanalyses of the previous 10 samples.
- Confirmation of a tentative identified analyte will occur on a secondary column. Any decision of the analyst to reject the tentative identification will be clearly documented in the data package, if necessary enlargements of chromatograms will be submitted.

7.1.2.4 Total Organic Carbon and Cation Exchange Capacity Analysis

Samples for TOC and CEC parameters will be analyzed according to the analytical procedures set forth in EPA SW-846 (see Table 7.1 for specific methods). The laboratory SOPs for sample preparation and analysis of each specific method are included in Appendix A of this QAPjP. The following QC criteria will be met:

- The laboratory will provide method-specific MS, LCS, and QC check sample recovery limits.
- A continuing calibration verification check standard (CCS) will be analyzed after 10 samples. QC samples like MS, LCS, or blanks count as samples. If a CCS exceeds the limit of ± 15 percent of the true value, the analyst must take corrective action, which may include reanalyses of all samples analyzed since the last previously acceptable CCS.

7.2 Field Screening Analytical Protocol

The standardization and QA information for field measurements are described in Appendix A of the TSP. Field screening procedures are discussed below.

7.2.1 Polychlorinated Biphenyl Soil Screening Analyses

The PCB soil screening will be used to assess the approximate concentrations of PCBs in FBH soil.

The PCB soil screening procedure includes the use of a spectrophotometer and commercially prepared PCB standards. The PCB screening procedure is specific for PCBs, and no interferences due

Analytical and Measurement Procedures

to other chlorinated compounds that may be present at the site are expected. Samples testing positive may be contaminated with PCBs and may be subject to confirmatory laboratory testing.

7.2.2 Volatile Organic Compounds Field Screening

Selected surface-soil samples will be screened for VOCs in the field. Results of the VOC screening will be used to decide whether subsequent laboratory VOC analyses are warranted. The screening test for VOCs will consist of an ambient temperature headspace analysis. A 16-ounce glass jar will be half filled with freshly sampled soil, sealed with a clean sheet of aluminum foil, agitated for at least 30 seconds, and allowed to come to ambient temperature (approximately 75°F). The tip of the OVA will then be inserted into the jar and the maximum concentration of VOCs in the headspace air measured. The samples should be warmed to 75°F prior to analysis. A OVA reading in excess of 50 ppm will instigate additional soil sample collection and laboratory analysis for VOCs.

7.3 Laboratory Sample Preparation Procedures

Standard QC and investigative samples will be prepared using method-specific procedures. Dilution of aqueous samples for organic analysis will be prepared using American Society for Testing and Materials (ASTM) Type II grade water, dilutions of aqueous samples for inorganic analysis be prepared using ASTM Type I grade water. Solvents used for soil extractions shall have their purity verified on a regular basis and extracts will be diluted using the same solvent that was used during extraction.

7.3.1 Sample Preparation of Aqueous Samples

Aqueous samples for the purpose of the Phase II RFI analytical program include groundwater. Groundwater samples to be analyzed for dissolved metals will be filtered during sample collection, before adding nitric acid or other preservatives. Metals analyses requiring field filtration are identified in the TSP. Silicon fiber or cellulose acetate filters with 0.45-micron pore size will be used for filtration. Samples to be analyzed for parameters other than metals will under no circumstances be filtered in the field. Aqueous samples to be analyzed for organics other than volatiles may be filtered in the laboratory using a compatible filter.

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Filter material is considered compatible if the filter material is not changed by the material being filtered, the material being filtered is not changed by the filter, and the filter material does not leach or adsorb the analytes of interest. Field blanks must also be filtered in the same manner as the investigative samples to isolate any carryover or constituent introduction that may be attributable to the filtration process.

Groundwater samples collected during the Phase II RFI will be analyzed in the field for pH, Eh, specific conductance, and temperature. Groundwater samples will be analyzed for pH and Eh using an Orion Model 250 (or equivalent) meter. Analysis of groundwater samples will necessitate the use of separate pH and Eh probes. Groundwater samples will be analyzed for conductivity and temperature using an Orion Model 123 Conductivity/Temperature Meter (or equivalent). Procedures for these analyses are provided in Appendix A of the TSP.

7.3.2 Sample Preparation of Soil Samples

Soil samples for the purpose of the Phase II RFI analytical program consist either of surface soil or subsurface soil. No chemical preservative will be used for soil samples. Soil samples submitted for VOC analyses will not be mixed in the laboratory prior to analysis. Subsurface-soil samples received by the laboratory, in 6-inch stainless-steel liners, will be subcored, and the outer 1 inch of soil from each end will be removed before selecting the method-specified aliquot for preparation and analysis. Each subsurface-soil sample will be dried using ASTM Procedure D2216-71 to estimate the moisture content. The percent moisture content will be calculated using the following equation:

$$\text{Percent moisture} = \frac{\text{sample wet weight} - \text{sample dry weight}}{\text{sample wet weight}} \times 100 \quad (7-1)$$

7.3.3 Toxicity Characteristic Leaching Procedure

Samples consisting of investigative-derived waste may be analyzed for RCRA toxicity. These samples will be extracted using the Toxicity Characteristic Leaching Procedure (TCLP) EPA SW-846

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Method 1311. The resulting extract will be analyzed for TCLP analytes using appropriate EPA-approved analytical methods.

8.0 INTERNAL QUALITY CONTROL CHECKS

This section presents a summary of field and laboratory QC check samples and procedures that will be analyzed as part of the RFI analytical program for the respective analytical methods.

8.1 Field Measurements

Investigative samples collected during the Phase II RFI will be collected following sampling protocol presented in Appendix A of the TSP. The assessment of field sampling precision and accuracy will be made through collection of field duplicates and field blanks. The frequency of field QC sample collection was described in Section 3.1 of this QAPjP.

8.1.1 Field Quality Control

QC procedures for measuring parameters in the field, including water level, pH, specific conductance, temperature, Eh, and organic vapor measurements of samples, will be used to calibrate the instruments, measure duplicate samples, and check the reproducibility of the measurements by taking multiple readings on a single sample or reference standard as described in Section 6.0 of this QAPjP.

8.1.2 Quality Control for Polychlorinated Biphenyl Screening

The PCB screening procedure will be performed onsite by HLA in a mobile laboratory. The QC for the PCB screening method will include the following:

- PCB Standards
 - Replicate standards will be analyzed as part of the method calibration. A valid PCB screening analysis will be indicated when the optical density difference of the replicate standards is 0.20 or less.
- Blanks
 - Method Blank - A sample of extraction solvent will be analyzed to evaluate reagent and equipment contamination. Method blanks will be analyzed after every 20 investigative samples, at a minimum. Additional system blanks will be analyzed whenever system contamination is suspected.
 - Field Blank - Soil will be collected as a field blank from areas with no known history of PCB contamination. One field blank will be analyzed for every 20 or fewer

Internal Quality Control Checks

investigative samples. Field blanks will be analyzed to assess possible chemical or physical interference.

- Duplicate Samples
 - Replicate Samples - Replicate soil samples will be analyzed to assess method repeatability. One replicate sample will be analyzed for every 5 or fewer investigative soil samples. The soil sample selected as a replicate will preferably be a sample testing positive for PCBs.
- Confirmatory Laboratory Analyses
 - Confirmatory Soil Samples - Soil samples screened for PCBs that tested positive, will be submitted to a USAEC-approved laboratory for confirmatory PCB analyses using EPA Method 8080. A minimum of 10 percent of the investigative soil samples will be submitted for confirmatory analyses.
- Matrix Spike Samples
 - FBH soil samples spiked with a known quantity of PCBs will be analyzed to assess field performance of the method and the analyst. One MS sample will be analyzed each day of PCB screening for every 20 or fewer investigative samples.

8.2 Laboratory Analyses Quality Control Checks

Internal quality control procedures for CLP RAS are specified in the respective SOWs for organic and inorganics analysis, or in the laboratory SOPs for herbicides, dioxins/furans, TPH, TOC, and CEC analysis (Appendix A of this QAPjP). These specifications include the types of QC checks required (method blanks, instrument blanks, reagent and preparation blanks, MS/MSDs, calibration standards, internal standards, surrogate standards, specific calibration verification check standards, and laboratory duplicate and replicate analysis), compounds, concentrations to be used, and the associated QC acceptance criteria for these QC checks. For a description of the specific QC requirements for the herbicide analysis, refer to the submitted SOP (Appendix A).

External (field) and internal (laboratory) QC samples as well as calibration curves and relevant reference materials will be used to monitor and quantify performance of analytical methods and field procedures. External QC samples are samples introduced into the sample train in the field to monitor the potential impact on reported results of sample collection activities, shipping, and

Internal Quality Control Checks

analytical performance. Internal QC samples are samples introduced into the sample train by laboratory personnel to monitor laboratory-induced contamination and analytical performance.

Calibration curves will be used as QC checks to ensure that analytical instruments are functioning properly at the required sensitivity. Relevant reference materials will be used to prepare the appropriate QC samples and calibration standards. The following subsections describe the requirements for external QC samples, internal QC samples, calibration curves, and reference materials.

8.2.1 External Quality Control Checks

External QC checks are samples introduced to the analytical stream during field operations to evaluate the impact of sampling activities and transport of investigative samples on the reported analytical results. External QC samples were described earlier in Section 3.1 of this QAPP, and include the following categories of samples and designated purposes:

- Natural Matrix Spike Samples
 - Native MS samples are created in the laboratory by adding known amounts and concentrations of method-specific target analytes into a prepared portion of a natural (representative sample medium) MS sample immediately before extraction or analysis. One MS and one MSD sample will be collected for every twenty investigative samples per sample matrix. For TAL metals, one MS sample will be collected.
- Duplicate Samples
 - Collocated Samples - Collocated samples are independent samples collected so they are equally representative of the parameter(s) of interest at a given point in space and time. When collected, processed, and analyzed by the same organization, these samples provide field and laboratory precision information for the entire measurement system, including sample acquisition, homogeneity, handling, shipping, storage, preparation, and analysis. They will be used to assess sample collection reproducibility and media variability. Field duplicates will be collected using the same techniques as those used to collect investigative samples. At least 1 field duplicate sample will be collected for every 10 investigative samples per sample matrix.
- Trip Blanks
 - Trip blanks pertain to VOC samples only. These blanks are prepared by the laboratory using analyte-free water before the sampling event in the actual sample bottles. Trip blank samples are kept with the investigative samples throughout the sampling event, then packaged for shipment to the laboratory for analysis with other samples. One trip blank should be included in each shipping container that contains samples

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for VOC analysis. At no time after preparation are trip blank sample bottles opened before they reach the laboratory for analysis.

- Rinse Blanks
 - Rinse blanks are defined as samples collected by rinsing analyte-free deionized water through sample collection equipment after decontamination and placing the collected water in the appropriate sample containers for analysis. These samples will be used to evaluate the adequacy of field decontamination procedures. Rinse blanks will be collected at the rate of one rinse blank per day per matrix per sampling equipment type.

8.2.2 Internal Quality Control Checks

The purpose of introducing internal QC check samples is to monitor day-to-day variations in routine laboratory analyses. It is essential that controls are initiated during and maintained throughout all steps, from sample preparation through sample analysis.

Internal laboratory QC samples provide both method control and individual sample control. Method control is provided, where applicable, through the analysis of method blanks, blank spiked samples, calibration standards, specific calibration verification check standards, and laboratory duplicate and replicate samples. Individual sample control is provided through the analysis of surrogate compounds and matrix spike compounds.

8.3 Quality Control Sample Documentation

To ensure the production of analytical data of known and documented usable quality, general guidelines with regard to quality control checks, quality assurance, and analyst training are presented below.

8.3.1 Quality Control Check Samples

QC samples will be assigned sample numbers by the laboratory sample custodian during the log-in process, as described in Section 5.2.1. Spiked QC samples will be prepared by a standards preparation specialist or the analyst responsible for the first step of an analytical method. The standards preparation specialist will be responsible for obtaining the appropriate volume/weight and type of

Internal Quality Control Checks

standard spike and matrix to be used. Spiking solutions and procedures must be identical to those specified in the written analytical method.

Method blank results will be reported uncorrected, as determined on the basis of the instrument calibration response factor. Blank contamination problems must be delineated by each laboratory. Unusual problems or problems that need immediate attention because they could impact the technical utility of the reported results must be discussed with the HLA Task QA Coordinator prior to the implementation of corrective action.

All sample analyses results must be within the calibration range of the analytical method. If necessary, the sample extract should be diluted or the original sample diluted to bring the analyte concentrations into the method calibration range. All samples must be reanalyzed immediately once it is discovered that method QC criteria have not been met, unless written approval is received from the HLA Task QA Coordinator.

8.3.2 Quality Assurance Program

The laboratory shall maintain a written QA/QC program which provides rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory's Quality Assurance Unit (QAU), which is independent of the operating departments.

The stated objectives of the laboratory QA/QC program are to:

- Check that all procedures are documented, including any changes in administrative and/or technical procedures.
- Check that all analytical procedures are conducted according to sound scientific principals and have been validated.
- Monitor the performance of the laboratory by a systemic inspection program and provide for corrective action as necessary.
- Collaborate with other laboratories in establishing quality levels, as appropriate.

- Check that all data are properly recorded and archived.

All laboratory procedures are documented in writing as SOPs which are edited and controlled by the QAU. Internal quality control procedures for analytical services will be conducted by the laboratory in accordance with the SOPs or the individual method requirements of the SOWs for organics and inorganics, as appropriate.

8.3.3 Analyst Training

It is the responsibility of the analytical laboratory to ensure that all laboratory personnel are qualified for their positions. Qualification includes any combination of education, training, or technical knowledge or experience, and this information must be documented. A new analyst using an established method is conditionally qualified until the first set of QC data is produced. The analyst is then considered either qualified or not, depending on the status of the QC data. If the QC data do not meet the QC requirements, corrective action (reanalysis or additional training) must be taken.

The LQAC must periodically inspect the laboratory to ensure that only qualified personnel are conducting the analyses. Each data package must be inspected by the LQAC to confirm that the sample analyses were performed under controlled conditions.

9.0 DATA REDUCTION, EVALUATION, AND REPORTING

This section presents a summary of methods for data reduction, evaluation, and reporting of data generated during the Phase II RFI.

9.1 Data Reduction

Data reduction is the process in which raw field and laboratory analytical data are converted to final results in the program reporting units. Data reduction is a multiphase task that affects the ultimate usability of program results and will include the following data processing steps:

1. Data collection (field or laboratory) and computation of results
2. Evaluation of preliminary results
3. Internal and external validation of results
4. Evaluation of the accuracy of reporting procedures
5. Plotting and spatial evaluation of analytical results
6. Identification and evaluation of project critical data points
7. Reevaluation of program results relative to initial program objectives
8. Generation of a report detailing data usability and technical utility

Reduced data are in final form until more work is performed or new information becomes available that modifies the effect of an element in the reduction process. A key element in data reduction is consistency. This requirement for consistency in data reduction is one of the primary purposes behind developing and implementing a QA program.

9.1.1 Field Data Reduction Procedures

Reduction of field measurement data will be performed in accordance with procedures described in Section 9.1.2. The validity of all data will be evaluated by checking calibration procedures used in the field and by comparing the data to previous measurements obtained at the specific site. The HLA Field Supervisor will summarize the data obtained from field measurements and will include this

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information in the field activities documentation report, which will be submitted to the HLA Task QA Coordinator for review.

9.1.2 Laboratory Data Reduction Procedures

Samples collected at FBH for Level IV analyses will be sent to the subcontract analytical laboratory. Data reduction, evaluation, and reporting for samples analyzed by the analytical laboratory will be performed according to specifications outlined in the appropriate CLP SOW for organics and for inorganics analyses, or EPA SW-846 methods (under an EPA CLP designation) for herbicides, dioxins/furans, TPH, TOC, and CEC analyses. Data validation will be performed in accordance with EPA's functional guidelines (EPA, 1994a, b). The laboratory will report analytical results in hardcopy and electronic formats as described in Section 9.4.

Reduction of analytical data will be performed in accordance with the following protocol. Bound logbooks with pre-numbered pages will be used for recordkeeping. In addition to the pre-numbered pages, each logbook or laboratory notebook will have a unique number for ease of identification. Additional documentation, such as chromatograms, will be referenced to the logbook or notebook, where appropriate. Loose sheets will not be used unless permanently affixed to the logbook. The use of bound books tends to result in a chronological sequence of data. Numbered pages encourage use of data in sequence and also aid in referencing data through a table of contents ordered according to time, type of analysis, type of sample, and/or identity of analyst.

Logbook entries shall be completed in ink. Corrections should be made by drawing one line through the incorrect entry, entering the correct information, initialling, and dating the change. A comment as to why the entry was crossed out should also be made. Complete information should be entered so that during a data review it can be determined what actions were performed when, by whom, and what the results were. At the end of each work shift, the analyst shall sign after the last entry is made.

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Validation is facilitated by requiring the sampler or analyst to date and sign each activity or analysis prior to the end of their work shift. This validation should be further strengthened by providing space in logbooks for the supervisor to initial.

For this project, the equations that will be used to reduce raw laboratory instrument output are included in the respective SOWs or SOP (Appendix A) describing the respective analytical procedures. Such formulae make pertinent allowance for matrix type. All calculations are checked by the appropriate laboratory supervisor at the conclusion of each operating day. Errors must be noted and corrections made, with the original notations crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

The data validation procedures presented in Section 9.2 specify the necessary documentation and technical criteria required to validate the data. When validated, the data will be evaluated with respect to PARCC and sensitivity parameters as described in Section 12.0. Upon completion of the data validation and evaluation of the data with respect to PARCC, HLA will develop and maintain summaries for each validated analytical lot. These reports, along with field activities documentation reports, will be prepared, summarizing the results obtained for samples collected during the RFI.

9.2 Data Validation

Technical data, including field data and results of laboratory sample analyses, will be validated to monitor the technical utility of reported results and the attainment of project DQOs. Procedures for validating field and laboratory data are described below.

9.2.1 Procedures Used to Validate Field Data

Field data to be evaluated include the raw data and supportive documentation generated from field investigations. Verifications of field procedures will be performed on 100 percent of the following field data:

- Field logbooks

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- Field investigation daily reports
- Field instrument readings and calibration data sheets
- Field logs of borings
- Field well completion data
- Groundwater sampling forms
- Sample tags
- Chain-of-custody forms
- Sample tracking records
- Surveying information
- Maps

Field measurements that could affect the quality of the data (such as temperature, pH, conductivity, or water level) will also be evaluated. Field data will be evaluated by the HLA Task QA Coordinator or a designated representative with respect to meeting project objectives. Field data will be evaluated by checking the procedures used in the field and comparing the current data to previous measurements.

The following areas will be addressed during evaluation:

- Sampling methodology
- Sample preservation
- Instrument selection and use
- Instrument calibration and standardization
- Instrument preventive and remedial maintenance
- Field deviations
- Sampling limitations

This evaluation will follow the initiation of the RFI data validation effort in response to the reported chemical results. The following verification procedures will be performed:

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- Chain-of-custody integrity check
- Appropriateness review of field methodologies
- Transcription, calculation, completeness, and accuracy checks of field data
- Analysis of field notes to evaluate bias

9.2.2 Procedures Used to Validate Laboratory Data

The analytical laboratory will perform analytical data reduction and in-house laboratory validation on all data under the direction of the LQAC. The LQAC will also be responsible for assessing data quality and advising appropriate section supervisors and the HLA Task QA Coordinator of (1) data that are considered to be "unacceptable" or (2) other problems with data quality that would affect the technical reliability of a result. Data validation by the laboratory will be conducted as follows:

1. Raw data produced by the analyst will be given to the respective section supervisor for review and comment.
2. The section supervisor will review the data relative to the required QC criteria outlined in this QAPP.
3. After acceptance of the raw data by the section supervisor, a report will be generated and sent to the LQAC for an independent review.
4. The LQAC will complete a thorough audit of 100 percent of the reported results for accuracy.
5. The LQAC and section supervisor will evaluate and communicate with the HLA Task QA Coordinator if reanalysis of any sample is required.
6. After acceptance of the preliminary results by the LQAC, transfer files or results reports will be generated, and the data sent to HLA.

The LQAC will ensure that a systematic process for evaluating data reduction and reporting at the laboratory is maintained. This evaluation process will consider the analytical sequence, calculation sheets, document control forms, blank data, duplicate data, recovery data for QC samples, and calibration standards. Data reports will be checked for legibility, completeness, correctness, necessary dates, initials, and signatures. Assessment of the analytical data will include checks for consistency by evaluating comparability of laboratory-generated duplicate analyses, comparability to previous data from the same sampling location (if available), adherence to method accuracy and

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precision criteria, transcription errors, and anomalously high or low parameter values. The results of this data checking process will be reported to the HLA Task QA Coordinator, verbally and in writing, if necessary, noting any discrepancies and their effect(s) on the data.

The LQAC will review at least the following analytical procedures and instrument performance criteria:

- Organic Compound Analyses
 - Data completeness
 - Sample holding time
 - GC/MS tuning and mass calibration (VOC, SVOC)
 - Instrument calibrations
 - Blank results
 - QC sample recoveries
 - Compound identifications
 - Compound quantifications
 - Spectral interpretation
 - Appropriate concentration units
 - Appropriate dilution factors and significant figures
 - Samples that exhibit carryover effects
 - Extraction efficiency
 - Inconsistency with known conditions or previous sampling results
 - Review of CLP laboratory flags applied to data
- Metals Analyses
 - Data completeness
 - Sample holding time
 - Instrument calibration
 - Blank results

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- Interference check sample analysis (for ICP)
- Analytical spike recoveries (for GFAA)
- Instrument detection limits (IDLs)
- QC sample recoveries
- Method of standard addition results
- Quarterly verification of instrument parameter report
- Appropriate concentration units
- Appropriate dilutions and significant figures
- Samples that exhibit carryover effects
- Inconsistency with known conditions or previous sampling results

Laboratory records and data package requirements will be checked to assess completeness of the data package.

9.2.2.1 Data Validation

An integral part of the RFI analytical program is the review and subsequent validation of the analytical data produced by the subcontractor laboratory. Although the primary responsibility for the review rests with the laboratory, HLA or the Army's designee will perform data reviews and validation independently from the laboratory. These procedures include the following:

- Laboratory audits - HLA will conduct two laboratory audits during the RFI analytical program. The laboratory audit procedures are described in Section 10.0, and the laboratory audit reports are described in Section 14.1.2.
- Electronic data review - HLA or the Army's designee will review electronic deliverables results to assess that the correct information regarding holding times, laboratory, installation, analyte names, and concentration units have been included in each file.
- Hardcopy data validation - HLA or the Army's designee will validate 100 percent of the data produced for each method during the RFI analytical program. The data validation procedures will be used by the HLA Task QA Coordinator or designated representative.

The EPA and IDEM have requested that 100 percent of the analytical results for the Phase II RFI be validated. Data validation will be conducted following the "USEPA Contract Laboratory Program

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National Functional Guidelines for Organic Data Review" (EPA, 1994a) and the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," Standard Data Validation Protocol (EPA, 1994b) or the most current versions available. For analytes and analyses not covered by these two documents, the general approach to performing data validation and qualifying data outlined in these documents will be used. However, specific acceptance criteria and QC limits will be taken from the analytical methodology for the non-CLP analysis. If specific criteria are not given in the methodology, professional judgment will be used to validate and qualify the data. One hundred percent of the lots will be reviewed to assess data package completeness and adherence to method-specific quality control criteria. In addition, approximately 20 percent of the calculations pertaining to method calibration and internal laboratory quality control results, and the raw data for the investigative samples will be reviewed to verify analyte identification and quantification. CLP data validation qualifiers will be added to the laboratory Form Is based on laboratory compliance with the method criteria, functional guidelines criteria, and professional judgment. The steps for the FBH RFI analytical data validation are outlined below:

1. Compile a list of all investigative samples.
2. Compile a list of all QC samples, including the following:
 - Rinse blanks
 - Field blanks
 - Trip blanks
 - Laboratory blanks
 - Duplicate samples (replicated or collocated samples)
 - Laboratory replicates
 - Matrix spikes
 - Matrix spike duplicates
3. Review laboratory analytical procedures and instrument performance criteria as follows for organic and inorganic analyses:

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- Organic Analysis
 - Technical sample holding time
 - GC/MS tuning and performance
 - Instrument calibration and performance
 - Blanks
 - Surrogate recoveries
 - MS/MSD recoveries
 - QC sample recoveries
 - Compound identification and quantitation
 - Tentatively identified compounds
 - System performance
 - Overall data assessment
 - Inorganic Analysis
 - Technical sample holding time
 - Instrument calibration
 - Blanks (laboratory and field QC)
 - Interference check sample analysis (for ICP)
 - Analytical spike recoveries (for GFAA)
 - ICP serial dilution
 - Laboratory control sample analysis
 - Matrix spike sample recoveries
 - Laboratory replicates
 - Sample result verification
 - Overall data assessment
4. Evaluate the integrity of the data as follows:
- Review chain-of-custody forms for completeness and correctness.
 - Review data for transcription, calculation, completeness, and accuracy errors.

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- Review laboratory analytical procedures, appropriateness, and instrument performance criteria.
5. Prepare a data summary that includes the following:
- Validated results
 - Media
 - Sample location and descriptions
 - Units of concentration
 - Definition of data qualifiers
6. Review data for potential inaccuracies, including the following:
- Unexpected results
 - Laboratory artifacts
 - Field-related artifacts
 - Unexpected spatial relationships
 - Samples in which dilution was necessary
 - Samples that may have been contaminated
 - Missed technical holding times

Laboratory records and data package requirements will be checked to assess completeness of the data package. The validation effort will be performed by personnel qualified and experienced in laboratory data validation.

Data validation qualifiers and definitions will be used as specified in EPA functional guidelines (EPA, 1994a, b). Qualifiers will be added to the validated data to indicate the data quality according to intended data use, and to ensure that data users are aware of limitations to, and quality of the data.

9.3 Statistical Evaluation

Statistical methods will be used to evaluate concentrations of target analytes in background and investigative site samples collected at FBH. Because some of the target analytes for this RFI occur naturally in FBH soil and water, it is necessary to distinguish between concentrations that represent

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releases of hazardous waste or hazardous constituents and background concentrations of target analytes.

In order to satisfy regulatory agency requests, HLA will use two approaches to assess the presence of elevated levels of potentially hazardous materials in environmental media. The first approach will use upper tolerance limits (UTLs), and the second will use analysis of variance (ANOVA). These approaches are discussed separately below.

9.3.1 Statistical Method for Analytical Data Evaluation Using Upper Tolerance Limits

HLA used UTLs to assess background concentrations of target analytes for the Phase I RFI. Elements of the statistical approach used for the Phase I RFI will be incorporated into the Phase II RFI statistical analysis with the follow revisions:

- Statistical analysis of background inorganic analytical data results (metals) will be performed, using methods described in the Final Phase I FBH RFI Report, for analytes that were detected in more than 50 percent of the respective background samples.
- Statistical analysis of Phase I background organic compounds analytical results will not be performed because individual organic compounds were infrequently detected in the background samples analyzed, and because the Phase II background soil samples will not be analyzed for organic compounds.
- HLA will discuss and compare concentrations of inorganic analytes and organic compounds detected at low concentrations in investigative samples, as necessary, to inorganic analytes and organic compounds detected in background samples.
- A table will be included in the Phase II RFI Report listing organic compounds detected in the Phase I background samples, that were accepted by IDEM, along with their respective concentrations. The Phase II RFI Report will summarize and discuss investigative sample analytical data. Phase II RFI analytical data will be included as an appendix in the Phase II RFI Report.

Figure 9.1 shows a decision logic diagram of the statistical evaluation of background analytical data using UTLs. The general logic and statistical methods used in this analysis are consistent with the EPA guidance documents, "Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities - Interim Final Guidance" (EPA, 1989) and the Addendum to the Interim Final Guidance (EPA, 1992).

9.3.1.1 Definition of Target Analyte Statistical Populations

The first step in evaluating the background analytical data will be to assess how the background analytical data should be grouped for subsequent calculation of UTLs and comparison to investigative analytical results. The Army will collect background soil samples for comparison to investigation site soil samples. A description of the background soil sampling program is provided in the Final TSP for the Phase II EI (HLA, 1996). For statistical analysis the background soil metals data will be grouped by soil association and sample depth. In addition, the Phase I background soil analytical results previously accepted by regulatory agencies will be combined with the Phase II background soil analytical results. The Phase I and Phase II background soil analytical results will be evaluated and compared (and the regulatory agencies consulted) before combining the data for statistical comparison to the data collected at investigation sites. Before calculating UTLs, the background soil metals data distribution will be evaluated.

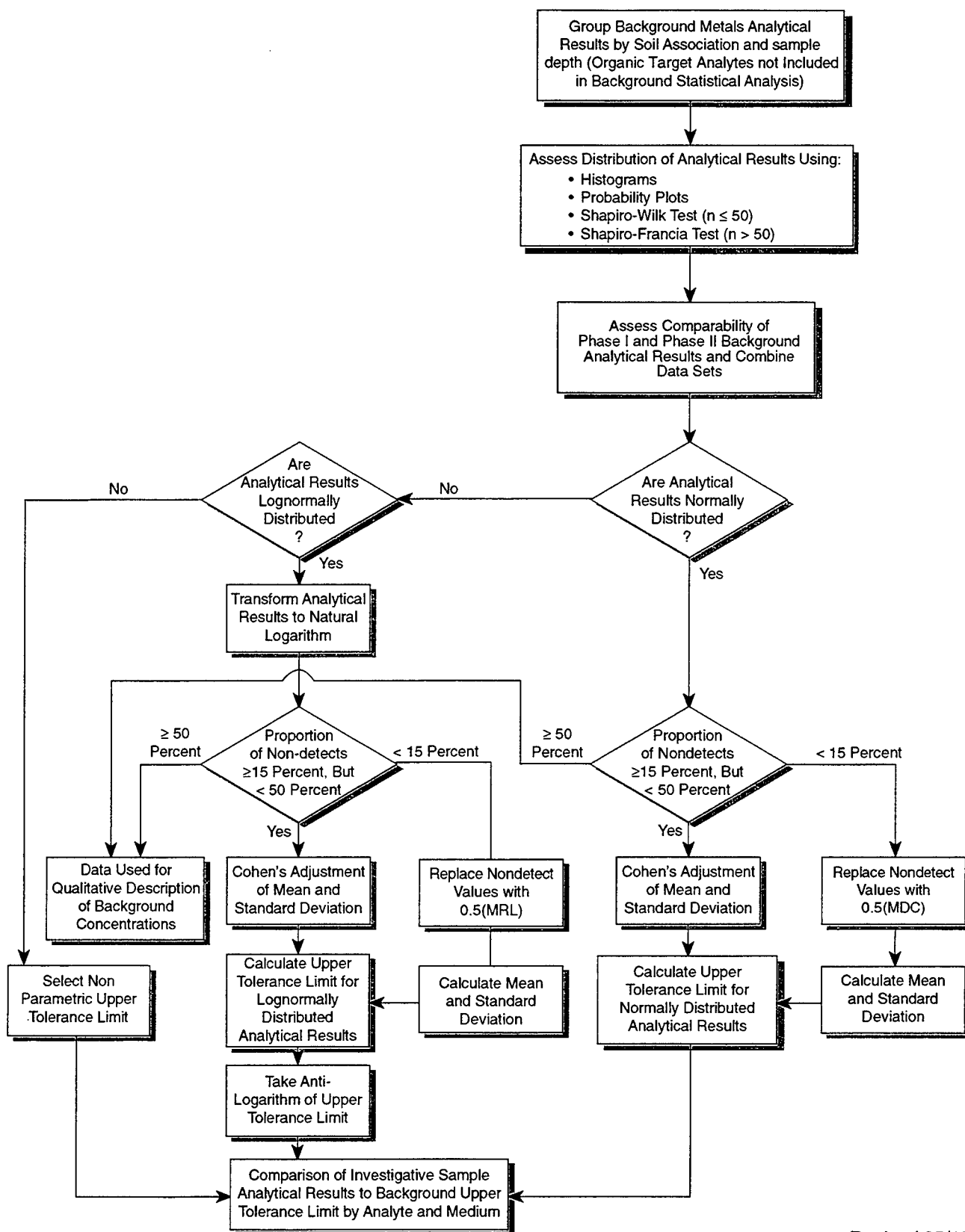
Evaluation of Statistical Distribution

The distribution of the background data for each analyte will be evaluated using probability plots, histograms, and the Shapiro-Wilk test for normality. Environmental data populations will be characterized as having normal, lognormal, or unknown distributions.

Histograms and Probability Plots

Histograms and probability plots are graphical methods that will be used to assess statistical distributions of each population defined for each medium and for each target analyte. Histograms show frequency of occurrence for each range of concentration. The data are assumed to be normally distributed if about two-third of the measurements fall within one standard deviation of the mean (EPA, 1989).

Probability plots are graphical methods that will be used to assess statistical distributions of each population defined for each medium and for each target analyte. A probability plot is constructed by plotting a data value on the x-axis, and plotting the proportion of observations less than or equal to



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Prepared for:
U.S. Army Environmental Center
Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
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Figure 9.1

Flow Chart for UTL Statistical Analysis of
Metals Background Analytical Results

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the observed value on the y-axis. The scale is constructed such that if the data are normally distributed, the plotted points will approximate a straight line. Interpretation of the linearity of the probability plot will be evaluated both visually and by calculating the correlation coefficient. Probability plots will be obtained using SYSTAT or another similar statistical software package. Probability plots and other graphics used in the analysis of the Phase II analytical results will be provided to regulatory agencies if requested.

Shapiro-Wilk Test for Normality

The Shapiro-Wilk test of normality is the recommended numerical test for checking data normality (EPA, 1992). The Shapiro-Wilk test is based on the premise that if the data are normally distributed, they should be highly correlated with corresponding quantiles taken from a normal distribution. This test can be performed for data sets with 3 to 50 samples. The procedure to perform this test is presented in the Addendum to the Interim Final Guidance (EPA, 1992).

Treatment of Nondetects

For the UTL calculation, analytical results below method reporting limits (MRLs) will be treated as follows depending on the percentage of nondetections.

- If the data are normal or lognormal and contain less than 15 percent nondetections, one-half the MDL will be used for the nondetect value, to calculate mean and standard deviation.
- If the data are normal or lognormal and contain between 15 and 50 percent nondetections, the MDL value will be used and the mean and standard deviation of the sample will be calculated using Cohen's Adjustment. Cohen's adjustment is described in the Addendum to the Interim Final Guidance (EPA, 1992).

9.3.1.2 Upper Tolerance Limit Calculation

UTLs will be calculated for each analyte within each medium, depending on the data distribution. Parametric UTLs will be calculated for a probability level (confidence factor of 0.95 and 95 percent coverage as described in the Interim Final Guidance [EPA, 1989]). For background data having an unknown distribution, a nonparametric UTL will be selected as the maximum detected value.

The UTL values for the target analytes detected in background samples will be used to compare to investigative site data. Analytes detected in investigative site samples that exceed their respective UTL will be identified as chemicals of concern. Analytes detected in investigative samples at concentrations equal to or less than the calculated UTL will be considered background.

9.3.2 Statistical Method for Analytical Data Evaluation Using Analysis of Variance

This section describes a second statistical analysis approach for evaluating FBH background and investigative site analytical results using one-way ANOVA or nonparametric ANOVA. This second statistical analysis approach has been reviewed and accepted by EPA Region V (CERCLA Program) and IDEM.

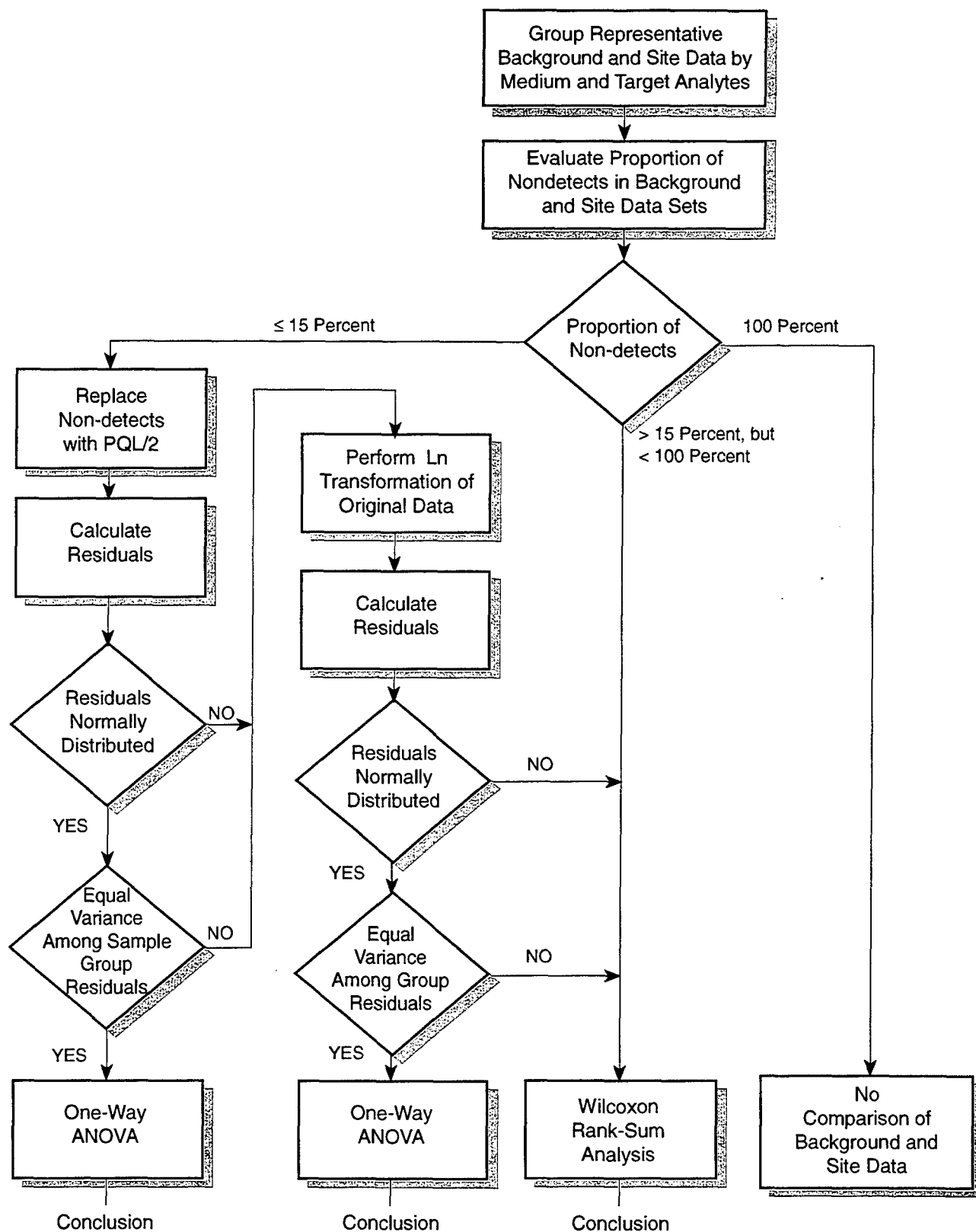
Accordingly, background and site data for the Phase II RFI will be reanalyzed as follows:

- Statistical analysis of background data will be based on central tendency using one-way ANOVA or nonparametric ANOVA.
- The statistical analysis will consist of a succession of two-group comparisons between analyte concentrations in background and investigative sites.
- Outlier analysis will not be performed.

The ANOVA procedures are consistent with methods described in "Statistical Analysis of Ground-water Monitoring Data at RCRA Facilities, Interim Final Guidance" (EPA, 1989). A flow diagram illustrating the major steps in the proposed approach is shown in Figure 9.2. Components of this approach are discussed below.

9.3.2.1 Definition of Target Analyte Statistical Populations

Statistical analysis will be initiated by evaluating the number of nondetections in background and investigative site data sets. The background data set will consist of the background data used previously for the UTL evaluation.



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Harding Lawson Associates
Engineering and
Environmental Services



Prepared for:
U.S. Army Environmental Center
Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
Marion County, Indiana

Figure 9.2

Statistical Analysis of Variance
Approach for Fort Benjamin Harrison
Environmental Investigation

Data Reduction, Evaluation and Reporting

The analytical data sets will be grouped in pairs for comparison during the assessment of proportion of nondetects. For example, background arsenic concentrations in surface soil will be compared to arsenic concentrations in surface soil at a specific investigation site. During the comparison, the background surface-soil arsenic analytical data and surface-soil arsenic analytical data from one investigative site will be grouped together. The number of nondetections in this group of surface-soil arsenic analytical data will then be assessed. This comparison will be repeated for each target analyte in each medium for each investigative site.

The proportion of nondetect values in the respective grouped data sets will dictate the subsequent type of statistical analysis performed. A data set having less than or equal to 15 percent nondetections will be evaluated for statistical analysis using the one-way ANOVA. Data sets having more than 15 percent, but less than 100 percent nondetections will be statistically analyzed using the Wilcoxon Rank-Sum Analysis. Data sets having 100 percent nondetects will not be statistically analyzed.

The data sets qualifying for one-way ANOVA analysis will be assessed for data distribution and variance. Probability plots and the Shapiro-Wilks test will be used to assess whether the data distribution of the analytical data are normal or lognormal, or fall under an alternate unknown distribution. An F-Test will be used to assess whether equal variance exists among the respective background analytical data, and the site analytical data being compared. Probability plots will be constructed using pooled residuals. Residuals are calculated as the difference between individual data values and the mean of its respective group, and are used to assess simultaneously the distribution or variance of more than one group of data.

9.3.2.2 Analysis of Variance

Data sets that are found to be normally or lognormally distributed with equal variances among the background and site data groups will be statistically analyzed using one-way ANOVA. Data sets that are not normally or lognormally distributed, or that do not exhibit equal variance among background

Data Reduction, Evaluation and Reporting

and site analytical data will be statistically analyzed using the Wilcoxon Rank-Sum Analysis. The method of calculating the one-way ANOVA is provided in the Interim Final Guidance (EPA, 1989). The method of calculating the Wilcoxon Rank-Sum Analysis is provided in the Addendum to the Interim Final Guidance (EPA, 1992).

The results of the one-way ANOVA statistical analysis and the Wilcoxon Rank-Sum Analysis will be used to help assess whether the sites being investigated exhibit elevated (greater than background) concentrations of target analytes. Elevated concentrations of target analytes may indicate a release to the environment. For the purposes of the Environmental Investigative proceeding under CERCLA for the transfer of Army property to other ownership, chemicals of concern will be assessed based on evaluation of results of the ANOVA statistical analysis.

9.4 Data Reporting

Sections 9.4.1 and 9.4.2 describe data reporting requirements for field and laboratory data.

9.4.1 Field Data Reporting

The HLA Field Supervisor will report field data principally by transmitting field measurements, recorded in field notebooks and copied, to the Task Manager on a weekly basis.

9.4.2 Laboratory Data Reporting

Analytical results for the FBH RFI will be reported in electronic form using the USAEC Installation Restoration Data Management Information System (IRDMIS) and in hardcopy form. Target analyte concentrations submitted for entry into the USAEC IRDMIS must remain unadjusted before being reported. Correction factors for sample moisture content and dilution factor will be incorporated into the final analytical values for storage in the NTAMs database system. Specific instructions are provided in the IRDMIS User's Guide (Potomac Research, Inc., 1995) regarding the proper method for coding of sample entries.

Data Reduction, Evaluation and Reporting

In reporting results, rounding to the correct number of significant figures occurs only after all calculations and manipulations are completed. The number of significant figures warranted by the analytical technique will be considered when reporting results. Premature rounding can significantly affect the final result. Data qualifiers applied during the data validation will be reported in the Final Phase II RFI summary report. However, because these data qualifiers are incompatible with the IRDMIS database structure, analytical data submitted to the NTAMs database system will not contain the data qualifiers applied during data validation.

9.4.2.1 Installation Restoration Data Management Information System Record and Group Checks

All data generated for FBH RFI will be stored in a computerized database format organized to facilitate data review and evaluation. Where possible, the data will be processed through the USAEC IRDMIS system. Data processed through IRDMIS exist in one of three levels as follows:

- Level 1 - Level 1 data are analytical data that have been initially accumulated and entered into the IRDMIS.
- Level 2 - Level 2 data have been loaded into the IRDMIS, validated by HLA or the Army's designee, and submitted by HLA or the Army's designee to USAEC for final processing.
- Level 3 - Level 3 data are analytical data that are stored after prior accumulation and verification. Level 3 data are generally considered unalterable.

Level 1 NTAM data loaded onto IRDMIS are run through a record check and then a group check. Every data point is checked using these two routines. The IRDMIS record check assesses the following:

1. Whether file name (such as CGW, CSW) and site type (BORE, WELL) combinations are valid
2. Validity of sampling program and technique, existence or absence of depth measurement
3. Whether any holding time violations occurred by comparing sample date, preparation/ extraction date, and analysis date
4. Whether test name, laboratory, installation, and prime contractor codes are valid
5. Whether concentration units match and are appropriate for the sample medium analyzed

Data Reduction, Evaluation and Reporting

The IRDMIS group check assesses whether all station identifications for the lot data exist in the map file for the appropriate installation. Specific criteria stored in the IRDMIS for record checks are based on the specific analytical method and on the current approval status of the laboratory performing the analysis.

If any errors are found in group and record checks that are not addressed on the lot cover sheet by the laboratory analysts or LQAC, the lot is returned to the LQAC so that the problem can be corrected. If changes to the analytical data are required, the lot is then resubmitted and, after revalidation, it is again processed through IRDMIS to ensure that any errors have been corrected.

After the data in a lot have successfully passed laboratory validation and IRDMIS record and group checks, a transfer file of the lot is created and sent to USAEC via modem. The data are again run through record and group checks by USAEC and, after passing the data checks, are elevated to Level 2.

The Level 2 data will be reviewed by USAEC. Any additional qualifiers resulting from their review will be entered into the IRDMIS. When USAEC is satisfied that the electronic data are correctly entered and qualified, the data will be elevated to Level 3.

9.4.2.2 Hardcopy Data Deliverables

The laboratory will prepare and retain full analytical and QC documentation as required by the CLP. Such retained documentation will include hard (paper) copy, and may also be in other storage media (e.g., magnetic tape). As needed, the laboratory will supply electronic and hard copy of the retained information. The laboratory will prepare and submit full analytical and QC reports to USAEC and HLA as requested in compliance with requirements of the CLP to include the following (as applicable):

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1. Narrative including statement of samples received, description of any deviations from RAS or SAS standard procedures, explanation of qualifications regarding data quality, and any other significant problems encountered during analysis.
2. Per sample, up to 20 extractable organic compounds not included in the RAS TCL will be reported as tentatively identified and quantified against the nearest internal standard.
3. An organic QA/QC report including CLP Forms I to X, and equivalent for the herbicide, dioxin/furans, and TPH analyses.
4. An inorganic QA/QC report including CLP Forms I to XIV, and equivalent for the TOC and CEC analyses.
5. Field and laboratory chain-of-custody documentation pertaining to each sample delivery group analyzed.
6. All associated raw data, as specified in the appropriate CLP SOW, or equivalent for the herbicide, dioxin/furans, TOC and CEC analyses.
7. Electronic data for submission to the PC IRDMIS NTAMS database.

The laboratory will report the data in the order in which the samples were analyzed within a given lot of samples, including associated QC data. The laboratory will provide the information in each analytical data package submitted as listed in Section 9.5.

9.5 Development and Usage of Document Control Procedures

Document control procedures are necessary to produce a litigation quality data package. A data package will contain the data necessary to support the results of one analytical method for one lot of samples.

In general, data will be maintained in two separate locations: the data package and the laboratory notebook(s). Records to be contained in the data package should include the information noted in Section 9.4.2.2, but are not limited to, the following:

- Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis.
- Original chromatograms, strip charts, or other instrument output
- Tabulated results of organic and inorganic analytes identified and quantified
- Original chain-of-custody form and carrier transmittal documents

Data Reduction, Evaluation and Reporting

- Hardcopy GC/MS output
- Expanded scale blowup of manually integrated peak(s)
- Data sheets or other preprinted forms used by the subcontractor laboratory
- Copies of relevant notebook pages. This should include preparation of standard solutions, calibration results, sample preparation/extraction, percent solid determinations, calculations, and other relevant comments.
- Analytical results for QC spikes, sample duplicate, initial and continuous calibration verifications of standards and blanks, standard procedural blanks, laboratory control samples, and ICP interference checks

Each data package should contain information related to one lot for FBH and a contents and approval checklist. This list should identify materials that must be placed into the data package. This list should also provide the reviewer's name(s), dates of review, space for comments, notes, and corrective actions.

It is the responsibility of the subcontractor laboratory to review data packages for both content and correctness (Section 9.2.2). Included in the data package should be a case narrative on the observations on the data contained in that package. This discussion shall include, but not be limited to, observed matrix effects, blank results, control problems, deviations from approved SOPs, and digressions from normal practices (i.e., manual integrations) and reasons thereof. The impact on the usability of the data shall be discussed. Explanations on the use of the applicable flagging codes and data qualifiers shall be provided.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the TSP and QAPjP. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

10.1 Field Audits

The following sections describe internal and external field audits.

10.1.1 Internal Field Audits

Internal audits of field activities (sampling and measurements) will be conducted by HLA and USAEC. The audits will include examination of field sampling records, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, and maintenance of QA procedures and chains of custody. These audits will occur at the onset of the project to verify that all established procedures are followed. Follow-up audits will be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the remediation. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation.

10.1.1.1 Internal Field Audit Responsibilities

USAEC or designated representative, as resources permit, and the HLA Task QA Coordinator or designated representative will audit field activities to evaluate sample identification, sample control, chain-of-custody procedures, field documentation, sampling operations, and handling and packaging procedures.

10.1.1.2 Internal Field Audit Frequency

USAEC or a designated representative, as resources permit, and HLA will audit field activities at least once at the beginning of the site sample collection activities. These audits will be unannounced to the field team. If problems are encountered during the initial audit, subsequent audits may be

conducted to correct deficiencies and verify that QA procedures are maintained throughout the project. Following the audit, preliminary results will be reviewed with the HLA Field Supervisor.

10.1.1.3 Internal Field Audit Procedures

The field audit will examine chain-of-custody records, field logbooks, and sampling operations. Field audit procedures are described in the following paragraphs. HLA will conduct field audits using the USAEC checklist provided in Appendix B.

Chain-of-Custody Records

The auditor will select a predetermined number of the chain-of-custody records to be audited in the field. The records will be reviewed to assess whether (1) the site identification site description, date, and time correspond to the sample label; (2) the parameters to be analyzed have been properly identified; and (3) all custody transfers have been documented and the date and time of transfer has been recorded. The auditor will also assess whether samples have been kept in custody at all times and have been properly and securely stored.

Field Logbooks

Field logbooks will be reviewed during the field audit to assess whether each is signed and all entries are dated. During field activities, notebooks will be kept in the possession of the sampling team leader. The project number, site name, date of receipt, and name of the person using the book will be recorded on each page.

All in situ measurements and field observations will be recorded in the notebook with all pertinent information necessary to explain and reconstruct sampling operations. Each page will be dated and signed by all individuals making entries on that page. The HLA Field Supervisor and the field team on duty will ensure that notebooks are available during all monitoring activities, and that they are safely stored at the end of each day's sampling activities and after the final day of field activities to maintain security. Any lost, damaged, or voided notebooks will be reported to the HLA Field Supervisor.

Performance and System Audits

Notebook entries must be legible, written in ink, and contain accurate and inclusive documentation of project activities. Language should be factual, objective, and free of speculation and inappropriate terminology. Entries made by individuals other than the person to whom the notebook was assigned must be signed and dated by the individual making the entry.

Photographs may be taken and must also be controlled. The auditor will review the field notebook to assess whether the photographs are properly documented. When slides or photographs are taken that show sampling sites or provide other documentation, they will be numbered to correspond to the notebook entries. The name of the photographer, date, time, site location, and site description will be entered sequentially in the notebook as photographs are taken.

The Field Supervisor's logbook will document the transfer of notebooks to the individuals who have been designated to perform specific field activities. All pertinent information will be recorded in these logbooks from the time each individual is assigned to the project until the project is completed. The auditor will review field notebooks for adherence to these procedures.

Sampling Operations

The auditor will review sampling operations to assess whether they are performed as stated in the TSP or as directed by the Field Work Activity Manager. The auditor will assess whether the correct number of samples were collected at the assigned locations and whether the samples were in appropriate containers and properly preserved. The auditor will also assess whether the required field measurements and QA checks have been performed and documented.

10.1.2 External Field Audits

An external audit will be conducted by EPA Region V Central Regional Laboratory and/or Central District Office.

Performance and System Audits

10.2 Laboratory Audits

Internal and external laboratory audits will be performed during the Phase II RFI. External laboratory audits will be performed by USAEC and HLA. External audits may be performed by the EPA Region V Central Regional Laboratory and/or Central District Office. The external audit may include system and performance audits.

The system audits will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedure, sample preparation and analysis, instrument operating records, etc. The performance audit will consist of sending performance evaluation (PE) samples to ESE for assessment of laboratory precision and accuracy. The results of the PE sample analyses are evaluated by EPA to ensure the laboratory maintains good performance. The following sections describe internal and external laboratory audits.

10.2.1 Internal Laboratory Audits

Internal laboratory audits are discussed below.

10.2.1.1 Internal Laboratory Audit Responsibilities

The internal laboratory audits will be performed by the USAEC Project Officer (as resources permit), the HLA Task QA Coordinator or designated representative, and the LQAC. Laboratory audits will focus on confirming the performance of the laboratory in implementing analytical requirements described in this QAPjP and the TSP.

10.2.1.2 Internal Laboratory Audit Frequency

Subsequent to project initiation, a USAEC representative will audit the analytical laboratory or sampling location to evaluate the effective implementation of the project QA program objectives. These audits will be conducted on a regular basis throughout the RFI analytical program. During this evaluation, analyses in progress will be open for inspection. Audit reports will be prepared by the USAEC project chemist and provided to HLA, the laboratory, and USAEC management as required.

Performance and System Audits

Audits may be scheduled or unscheduled, and the frequency of the audits will be increased if program inconsistencies demand a closer level of external control.

Internal audits will be conducted by the LQAC and the HLA Task QA Coordinator or designated representative on a quarterly basis. These audits will primarily target discrepancies observed during USAEC audits and include a detailed review of laboratory and field procedures using a step-by-step approach. Audits conducted by the HLA Task QA Coordinator will also include personnel training status, records, QC data, calibrations, and conformance to SOPs. Internal audit procedures will be performed in such a way that all phases of analysis are reviewed sequentially in detail.

10.2.1.3 Internal Laboratory Audit Procedures

Laboratory audits conducted by HLA will include a systematic review of laboratory facilities, equipment, training procedures, recordkeeping, data validation, data management, reporting, and QA checks. During this review, the LQAC and HLA Task QA Coordinator or designated representative will carefully review the standard preparation procedures, calibration and tuning logbooks, raw data collection, and confirmation steps. The LQAC and HLA Task QA Coordinator or designated representative will attempt to identify laboratory discrepancies with the QA program, overall deficiencies, and inappropriate laboratory practices. Based on results of the audit, corrective action will be suggested. If the corrective action required is sufficiently serious, project work will be stopped until corrective action has been performed and a work restart order is authorized by the HLA Task QA Coordinator.

Audits will be performed before and during the analysis of project samples. Audits may be announced or unannounced. Copies of audit reports will be supplied to USAEC for review and comment. Corrective action forms or requirements, in addition to the formal audit report, will be supplied to the LQAC.

Performance and System Audits

Performance evaluation samples may be sent by the HLA Task QA Coordinator to the analytical laboratory for analysis. These samples will be used to evaluate the performance of the laboratory.

10.2.2 External Laboratory Audits

An external audit will be conducted by the EPA Region V Central Regional Laboratory at least once before initiating the sampling and analysis activities. These audits may or may not be announced and are at EPA's discretion.

External laboratory audits will include, but not be limited to, review of laboratory analytical procedures, laboratory onsite audits, and/or submission of performance evaluation samples to the laboratory for analysis following EPA Region V procedures.

11.0 PREVENTIVE MAINTENANCE

Preventive maintenance will be performed on both field equipment and laboratory instruments.

11.1 Field Instrument Preventive Maintenance

A variety of instruments, equipment, and sampling tools will be used to (1) collect data and samples and (2) monitor field conditions. Correct calibration, maintenance, and use of instruments and equipment are required to ensure the quality of field data collected.

11.1.1 Inspection

All instruments and equipment purchased or used will be inspected to ensure that each item meets and performs to manufacturers' specifications and project specifications. Instruments meeting these requirements are given an ID number and made available for field use. Instruments and equipment not meeting program requirements are labeled and are withheld from field use. Such instruments and equipment are not available for use until they can be modified or repaired to meet field requirements.

11.1.2 Operating Procedures

The calibration, maintenance, and operating procedures for field instruments, equipment, and sampling tools are documented in the owner/operator manuals supplied by the manufacturer. These manuals provide manufacturers' instructions and include specifications and criteria for calibration, maintenance, and operation.

11.1.3 Field Equipment Calibration

General field equipment calibration procedures were presented in Section 6.1 and are summarized in Table 11.1.

11.1.4 Maintenance

Each item of equipment used in field activities is maintained to specifications presented by the manufacturer. The Field Work Activity Manager will be responsible for ensuring that routine

Preventive Maintenance

maintenance is performed, that tools and spare parts to conduct routine maintenance are available, and that procedures for maintaining instruments are consistent with manufacturers' operations manuals. Instruments will be calibrated to correct specifications following maintenance to ensure proper completion of the maintenance procedure.

A record of maintenance, including a description of specific activities performed, will be made in the field logbook, which is kept with the instrument. Data recorded in the logbook are similar to data recorded for calibration.

If the equipment or instrument cannot be maintained to the manufacturer's specifications or cannot be properly calibrated, it will be returned to the manufacturer or other repair facility for maintenance and/or repair. When returned from the manufacturer, the instrument will be checked for compliance to project specifications before being returned to routine field use.

11.2 Laboratory Instrument Preventive Maintenance

All laboratories participating in the CLP are required, under the CLP RAS SOW (OLM03.1 or OLC01.0) for organics and SOW (ILM03.0 or OLC01.0) for inorganics, to have SOPs for preventive maintenance for each measurement system and required support activity. All maintenance activities are required to be documented in logbooks to provide a history of maintenance records.

Every instrument or measuring device must be assigned a maintenance log number or record number and labeled. This label will be used by maintenance personnel and will include a description of the instrument, manufacturer, model number, serial number, date of last calibration or maintenance, signature of maintenance personnel, and the date when the next service check is required. A maintenance or calibration logbook associated with the instrument must be kept in the area where the measuring device is operated.

**Table 11.1: Routine Preventive Maintenance Procedures and Schedules
for Field Instruments**

Instrument	Maintenance Procedures/Schedules
Water-level Measurements	
Electrical sounder calibration:	Check against steel surveyor's tape before use.
Graduated steel tape calibration:	Manufacturer-supplied temperature correction will be applied if applicable for field conditions.
Pressure transducer calibration:	Factory calibration will occur once; in-house calibration check with water columns will occur before aquifer tests; and weekly field checks against steel tape or electrical sounder will occur during use.
Eh Measurements	
Digital Eh meter (Orion Model 250 or equivalent)	Zero Eh meter following manufacturer's instructions. Inspect electrode before each use for cracks or other signs of wear that may affect performance.
pH Measurement	
Digital pH meter calibration (Beckman Model 021 or equivalent):	Factory-supplied or laboratory-supplied buffer solutions will be renewed daily; instrument calibration will be checked before each measurement; temperature correction will be applied during measurement; and batteries will be checked daily.
Specific Conductance	
Electric conductivity meter calibration (YSI Model 51B or equivalent):	Factory calibration will occur annually; calibration will occur before each use using laboratory-supplied potassium chloride standard; temperature correction will be applied during measurement; and batteries will be checked daily.
Water Temperature	
Mercury thermometer calibration:	Factory calibration will occur once and will be checked at least biannually.
Temperature meter calibration:	Calibration will occur weekly against a mercury thermometer. Thermometers will be inspected daily for cracks or gaps in mercury.

Chemical calibrations are not considered absolute calibrations and are therefore not addressed under standard maintenance procedures. Chemical calibrations are useful as indicators of instrument problems or calibration standard degradation and may therefore be used to qualitatively identify nonroutine or unscheduled maintenance requirements.

Absolute calibration procedures must be kept with the instruments that require this type of calibration, including the following types of laboratory apparatuses:

- Instrument recording units, such as chart recorders and analytical balances
- Flow controllers or instruments that must remain constant to ensure instrument stability, such as flow controllers on fume hoods and GC gas supplies
- Temperature sensors of all types, such as thermometers and heat sensors in drying ovens
- Syringes, pipetors, and pumps used to introduce standards, solvents, and investigative or QC samples

Instruments or measuring devices that have not received maintenance or routine calibration must not be used to perform project-related work. If an instrument requires absolute calibration based on historical or manufacturer's recommended guidelines, it will be removed from service until such maintenance can be performed. In such cases, a physical label must be placed in an obvious location on the instrument to prevent accidental use of uncalibrated equipment. Such instruments should be removed from locations in which accidental use could easily occur.

Calibration of critical path instrumentation and routine instrument maintenance must be traceable through instrument tags or maintenance logbooks. Absolute calibrations must be performed using the appropriate and accepted absolute standard reference. For balances, NIST-certified Class "S" weights should be used to calibrate instrumentation. Use of in-house Class "S" weights is acceptable for daily calibration, but a yearly certification by an external inspector should also be performed. Similarly, an NIST-certified thermometer should routinely be used to calibrate temperature gauging devices. In all cases when calibration is performed, it must be easily traceable in the maintenance

logbooks. Instruments that do not require certified calibration, such as instrument flow rates, should be checked routinely. For example, GC/MS and GC flow rates should be checked daily or on a regular basis to ensure consistency of analytical conditions.

Each laboratory must maintain a standard procedures manual for preventive maintenance of critical and routinely used instrumentation. Each laboratory must provide these standard procedures to each analyst so in the event that instrument maintenance is required, the appropriate source of assistance is readily available. Each laboratory must employ a qualified maintenance person or be covered by a maintenance contract for project-required instruments.

The maintenance program employed by a laboratory should be targeted at minimizing instrument downtime. The substitution for calibration protocol described in this document must be reviewed and approved by USAEC before the initiation of program activities.

11.3 Record Keeping

Every instrument used in the program will have an associated instrument logbook. This logbook will be kept with the instrument and will not, under any circumstances, be removed from the instrument location. This logbook will be bound and project-specific, if possible. Daily instrument operations will be recorded in this logbook to allow reconstruction of the daily operating sequence. Each entry to this logbook will be signed by the analyst responsible for that particular injection or maintenance procedure. Instrument activities, such as reanalyses and instrument maintenance time, will be recorded in this logbook. Under no circumstances will previous entries be deleted or unauthorized new entries be added to this logbook. Each entry for sample analysis will include, but not be limited to, the following:

- Date of analysis
- Test name
- Project ID

- Sequential number
- Associated calibration and method blank/QC samples
- Analysis time of injection
- Amount of sample injected
- Comments concerning instrument performance
- Analyst's signature

When automated data acquisition systems are used, reference to the data file for each standard or sample will be recorded. Hardcopy output (e.g., chromatograms and integrator tapes) from instruments will be labeled with analyst's name, analysis time, test name, sample number, reference to the calibration curve used for quantification, and reference to the logbook where analytical activities were recorded. The identity of chromatographic peaks will also be noted. The hardcopy output will be maintained with the lot data packages. An individual instrument maintenance logbook will be permanently assigned to each instrument and will not be turned over to USAEC after project completion.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, AND SENSITIVITY

This section includes procedures for assessing data quality in terms of completeness and sensitivity.

12.1 Field Measurements

Field data will be assessed by the site QC Officer. The site QC Officer will review the field results for compliance with the established QC criteria that are specified in the QAPP and TSP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed based on the reproducibility of multiple measurements of a single sample. Data completeness will be calculated using Equation 12-1.

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \quad (12-1)$$

12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity as follows:

12.2.1 Precision

Precision of laboratory analysis will be assessed by HLA comparing the analytical results between MS/MSD for organic analysis and laboratory duplicate analyses for inorganic analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analysis using Equation 12-2.

$$\text{RPD} = \frac{S - D}{(S + D)/2} \times 100 \quad (12-2)$$

where:

S = First sample value (original or MS value)

D = Second sample value (duplicate or MSD value)

Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

12.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section 3.0 of this QAPjP using the analytical results of method blanks, reagent/preparation blank, MS/MSD samples, field blanks, and bottle blanks. The percent recovery (%R) of MS samples will be calculated using Equation 12-3.

$$\%R = \frac{A - B}{C} \times 100 \quad (12-3)$$

where:

A = The analyte concentration determined experimentally from the spiked sample

B = The background concentration determined by a separate analysis of the unspiked sample

C = The amount of the spike added

12.2.3 Completeness

The data completeness of laboratory analytical results will be assessed for compliance with the amount of data required for decision making. Completeness is calculated using Equation 12-1.

12.2.4 Sensitivity

The achievement of MDLs depends on instrument sensitivity and sample matrix effects. Therefore, it is important to monitor instrument sensitivity through constant instrument performance checks in order to ensure the data quality. Instrument sensitivity will be monitored through the analysis of method blanks, calibration check samples, laboratory control samples, etc.

13.0 CORRECTIVE ACTION

This section discusses corrective actions to be followed in the laboratory and the field to ensure the integrity of analytical data generated for the RFI.

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-quality control performance, which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All proposed and implemented corrective action should be documented in the regular QA reports to management. Corrective action should only be implemented after approval by the USAEC Project Officer or designee and the HLA Task Manager. If immediate corrective action is required, approvals secured by telephone from the HLA Task Manager should be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the HLA Task Manager, who in turn will notify the EPA RCRA Project Coordinator. If the problem is analytical in nature, information on these problems will be promptly communicated to the EPA, QA Section. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established QC procedures in the QAPjP or TSP will be identified and corrected in accordance with the QAPjP. The HLA Task Manager or designee will issue a nonconformance report for each nonconformance condition.

13.1 Field Corrective Action

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances, or suspected deficiencies of any activity or issued document, by reporting the

Corrective Action

situation to the HLA Task QA Coordinator or designee. This individual will be responsible for (1) assessing the suspected problems, in consultation with the HLA Field Supervisor and (2) making a decision, based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the HLA Task QA Coordinator.

The HLA Field Supervisor will be responsible for assuring that corrective actions for nonconformances are initiated by:

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining the disposition or action to be taken
- Maintaining a log of nonconformances
- Reviewing nonconformance reports and corrective actions taken
- Assuring nonconformance reports are included in the final site documentation in project files

If appropriate, the HLA Field Supervisor will ensure that additional work that is dependent on the nonconforming activity is not performed until the corrective actions are completed.

Corrective action for field measurements may include the following:

- Repeat the measurement to check the error
- Check for all proper adjustments for ambient conditions such as temperature
- Check the batteries
- Recalibration
- Check the calibration
- Replace the instrument or measurement devices
- Stop work (if necessary)

Corrective Action

The HLA Task Manager or designee is responsible for all site activities. In this role, the HLA Task Manager, at times, is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible person notifies the HLA Task Manager of the anticipated change and implements the necessary changes after obtaining the approval of the HLA Task Manager.

Corrective action in the field may be needed when the sampling network is changed or sampling procedures and/or field analytical procedures require modification due to unexpected conditions.

During the initiation of field activities, the HLA Task QA Coordinator will identify and immediately enforce corrective action onsite. This action includes correcting sampling procedures that violate sample integrity, resampling to replace affected samples, repackaging samples for shipment, and reCompleting chain-of-custody forms or field measurement forms. Samples involved with corrective action performance will be documented along with the nature and extent of the required action. The USAEC Project Officer will approve and HLA will implement any resampling that is necessary as part of a field corrective action. All corrective activities will be recorded in the project field logbook, and a copy of all corrective action notes will be provided to the HLA Field Supervisor and HLA Task Manager. If corrective actions are insufficient, work may be stopped by the EPA RCRA Project Coordinator.

13.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analysis. Conditions such as broken sample containers, multiple phases, low/high pH readings, and/or potentially high concentration samples may be identified during sample log-in or prior to analysis. Following consultation with laboratory analysts and section leaders, it may be necessary for the LQAC to approve the implementation of corrective action. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures.

Corrective Action

These conditions may include dilution of samples, additional sample extract cleanup, or automatic reinjection/reanalysis when certain QC criteria are not met.

Corrective actions will be implemented whenever (1) method-specific QC criteria are not met (criteria will be included in methods when available), (2) the project-specific QC samples (Section 8.0) do not meet project objectives, or (3) anyone associated with the project notices that a problem exists which could jeopardize the integrity of investigative sample results. Corrective action or stop-work memoranda may be authorized by the USAEC Project Officer, the HLA Task Manager, the HLA Task QA Coordinator, the LQAC, or the laboratory section supervisor. Analysts who suspect that an out-of-control situation may exist are obligated to inform their section supervisor of the problem before stopping work. If previously reported data are affected by a situation requiring corrective action or if the corrective action will impact the program budget or schedule, the action should directly involve the HLA Task Manager and the USAEC Project Officer.

Corrective actions are of the following two kinds:

1. Immediate, to correct or repair nonconforming equipment and systems. The need for such an action will most frequently be identified by the analyst as a result of calibration checks and QC sample analyses.
2. Long term, to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include the following:
 - Training staff in technical skills or in implementing the QA program
 - Rescheduling laboratory routines to ensure analysis is within allowed holding times
 - Identifying vendors to supply reagents of sufficient purity
 - Revising the contractor QA system or replacing personnel

For either immediate or long-term corrective actions, the steps comprising a closed-loop corrective action system follow:

1. Define the problem.

Corrective Action

2. Assign responsibility for investigating the problem.
3. Investigate and determine the cause of the problem.
4. Determine a corrective action to eliminate the problem.
5. Assign and accept responsibility for implementing the corrective action.
6. Establish the effectiveness of the corrective action and implement the correction.
7. Verify that the corrective action has eliminated the problem.

Depending on the nature of the problem, the corrective action may be formal or informal. In either case, occurrence of the problem, corrective action, and verification that the problem has been eliminated must be documented. In addition, if the corrective action results in the preparation of a new standard or calibration solution, then a comparison of the new solution versus the old solution needs to be performed and the results supplied with the weekly QC submittal as verification that the problem has been eliminated.

Corrective action will be implemented depending on the scope of the action required and the type of control being enacted. The following sequence of steps should be followed by laboratory personnel or management when an out-of-control situation becomes apparent:

1. If a problem is identified that potentially affects analytical results, analyses should be discontinued until the analytical sequence is demonstrated to be reliably stabilized.
2. When the problem has been identified, HLA's Task QA Coordinator should be contacted and informed of the possible net effect of the problem on reported results. This information is especially important when errors in calculations or improper processing are involved and the net effect on previously analyzed samples may be unknown.

In all cases when QC criteria are not achieved, affected samples should be immediately reanalyzed, whenever possible, within the specified holding time.

The LQAC is responsible for documenting the corrective actions performed during a project. The standard form (Figure 13.1) will be used to document all critical dates and information needed to trace the analytical results that may have been affected by any such action. All corrective actions

Corrective Action

will be coordinated among the LQAC, the Laboratory Analytical Task Manager, and the appropriate analytical section supervisors. When a problem has been identified, the complete analytical sequence should be reviewed to evaluate the true source of the problem. For example, all data processing procedures, calculations, blank results, calibrations, tuning parameters, interference checks, overall instrument sensitivity, logbook entries, standards or sample preparation, chromatograms, quantification reports, and digestions should be checked and noted in the corrective action report. All corrective actions will, in this way, document the circumstances surrounding a specific type of analytical problem for future investigators. Corrective action reports should be selectively completed for each project and included in the final evidence file.

13.3 Corrective Action During Data Validation and Data Assessment

HLA or USAEC may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent on the ability to mobilize the field team, whether the data to be collected are necessary to meet the required QA objectives (e.g., the holding limits are not exceeded, etc.). When the HLA data assessor identifies a corrective action situation, it is the HLA Task Manager who will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the HLA Task QA Coordinator.

Quality Assurance/Quality Control Corrective Action Record

Laboratory Name _____ USAEC ID No. _____

Problem Analyte(s) _____

Quality Assurance Comments:

LQAC _____ Date: _____

Management Action:

Signature: _____ Date: _____

Analyst Response

Signature: (use back of form if needed) _____ Date: _____

Quality Assurance Approval:

QA Advisor: _____ Date: _____

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Harding Lawson Associates
Engineering and
Environmental Services



Prepared for:
U.S. Army Environmental Center
Aberdeen Proving Ground, Maryland

Fort Benjamin Harrison
Marion County, Indiana

Figure 13.1
Corrective Action Record

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

In addition to the audit reports submitted to the Site Manager in accordance with Section 12.0 of this QAPjP, a monthly progress report is submitted to the EPA Remedial Project Manager (RPM) and HLA that addresses all QA issues. The Final RFI Report will contain QA sections that summarize data quality information collected during the project and will be submitted to USAEC during the RFI.

14.1 Contents of Project Quality Assurance Reports

Laboratory QA reports will describe details of progress and any concerns with chemical analysis activities. Laboratory audit reports will describe the laboratory activities and systems audited, including any audit findings. Field QA reports will describe notable field-related events or practices that may affect physical or chemical results, and field audit reports will describe the field activities audited and any audit findings.

14.1.1 Laboratory Quality Control Reports

Normal submissions to USAEC will include IRDMIS data submissions and audit reports. During the course of laboratory analyses, method procedural or control problems will be documented, explained, and reported to the HLA Task QA Coordinator. Corrective measures and reanalysis of samples must also be reported as indicated above. The HLA Task QA Coordinator, in conjunction with the LQAC and HLA Task Data Manager, will provide tabulation of QC sample data, as well as specific observations delineating the analytical method control, in an appendix to the Phase II RFI Summary Report. These observations will include the following:

- Analytical results of laboratory quality control, including method blanks, surrogate recoveries, and matrix spike recoveries
- Possible effects of results detected in method blanks on sample results
- Unique matrix characteristics of the environmental samples

Quality Assurance Report to Management

During the analytical effort, if a process or analysis is found to not be in control (i.e., method-specific QC criteria not met), a discussion of the problem, including the following information, will be submitted by the LQAC to the HLA Task QA Coordinator:

- Basis for judging a method to be out of control
- Investigation of the out of control situation
- Actions taken to bring the method back in control
- Actions taken to prevent reoccurrence of the out-of-control situation
- Disposition of the data acquired while the method was out of control

14.1.2 Laboratory Audit Reports

External and internal laboratory audit reports will be prepared during the RFI analytical program. External laboratory audit reports will be prepared by EPA as discussed in Section 10.2. Internal audit reports will be prepared by the LQAC and the HLA Task QA Coordinator.

These reports will document the activities and systems audited, any deficiencies, discrepancies, or inappropriate practices identified, and any corrective actions implemented during the audit.

In addition to the requirements for the LQAC audit reports, audit reports prepared by the HLA Task QA Coordinator will include a description of any required corrective actions implemented as a result of the audit findings.

14.1.3 Field Quality Assurance Reports

Field QA reports will be prepared and maintained in the RFI files. Field QA reports will provide a detailed review of site conditions experienced during the performance of task activities. The emphasis of these reports will be on identifying field-related activities that may have affected physical or chemical results. Field QA reports will be prepared by the HLA Field Supervisor.

Quality Assurance Report to Management

14.1.4 Field Audit Reports

Field audit reports will be prepared by the HLA Task QA Coordinator or designated representative.

Field audit reports will present a description of the activities and systems audited and any corrective actions or deficiencies noted.

14.2 Frequency of Quality Assurance Reports

Laboratory QA reports will be provided to the USAEC Chemistry Branch upon request. Laboratory and field audit reports will be prepared within 30 days of completion of the audit.

14.3 Individuals Receiving/Reviewing Quality Assurance Reports

Laboratory QA reports will be submitted by the LQAC to the USAEC Chemistry Branch upon request.

Field QA reports will be submitted to the HLA FBH Task Manager. Laboratory audit reports will be submitted to the HLA Task QA Coordinator, HLA Task Manager, and the USAEC Project Officer.

Laboratory audit reports prepared by the HLA Task QA Coordinator will be provided to the USAEC Project Officer, the Laboratory Analytical Task Manager, and the LQAC. Field audit reports will be provided to the HLA Task Manager, Field Supervisor, and the USAEC Project Officer.

15.0 GLOSSARY

%R	Percent recovery
2,4-DNT	2,4-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
ACE	U.S. Army Corps of Engineers
ACMs	Asbestos-containing materials
ANOVA	Analysis of variance
Army	U.S. Department of the Army
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BRAC	Base Realignment and Closure
CAS	Chemical Abstracts Service
CCB	Continuing calibration blank
CCS	Continuing calibration verification check standard
CCV	Continuing calibration verification
Cd	Cadmium
CEC	Cation exchange capacity
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CMS	Corrective measures study
COC	Chain of custody
COE	U.S. Army Corps of Engineers
COR	Contracting Officer's Representative
Cr	Chromium
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CVAA	Cold vapor atomic absorption

Glossary

DDD	2,2-bis(Para-chlorophenyl)-1,1-dichloroethane
DDE	2,2-bis(Para-chlorophenyl)-1,1-dichloroethene
DDT	2,2-bis(Para-chlorophenyl)-1,1,1-trichloroethane
DFTPP	Decafluorotriphenyl phosphine
DQO	Data quality objective
DRMO	Defense Reutilization and Marketing Office
Eh	Oxidation reduction potential measured in millivolts
EI	Environmental investigation
EPA	U.S. Environmental Protection Agency
ESE	Environmental Science and Engineering, Inc.
FBH	Fort Benjamin Harrison
GC/MS	Gas chromatography/mass spectrometry
GC	Gas chromatograph
GFAA	Graphite furnace atomic absorption
H ₂ SO ₄	Sulfuric acid
HCl	Hydrochloric acid
Hg	Mercury
HLA	Harding Lawson Associates
HNO ₃	Nitric acid
ICP	Inductively coupled argon plasma
ICV	Independent calibration verification
ID	Identification
IDEM	Indiana Department of Environmental Management
IDL	Instrument detection limit
IRDMIS	Installation Restoration Data Management Information System
IRMs	Interim reference materials
LQAC	Laboratory Quality Assurance Coordinator

Glossary

MDL	Method detection limit
mg/l	Milligrams per liter
mg/kg	Milligrams per kilogram
ml	Milliliter
MRD	Missouri River Division
MRL	Method reporting limit
MRP	Management/Resource Plan
MRR	Method Reporting Range
MS	Matrix spike
MSD	Matrix spike duplicate
Na ₂ S ₂ O ₃	Sodium thiosulfate
NaOH	Sodium hydroxide
NBS	National Bureau of Standards
Ni	Nickel
NIST	National Institute of Standards and Technology
NTAM	Non-THAMA approved method
OSHA	Occupational Safety and Health Administration
OVA	Organic vapor analyzer
PA	Preliminary assessment
PAH	Polynuclear aromatic hydrocarbon
PARCC	Precision, accuracy, representativeness, completeness, and comparability
Pb	Lead
PCB	Polychlorinated biphenyl
PE	Performance evaluation
PEM	Performance evaluation mixture
pH	Negative log ₁₀ of the hydrogen ion concentration
PID	Photoionization detector

Glossary

PQL	Practical quantitation limit
QA	Quality assurance
QAPjP	Quality Assurance Project Plan
QAU	Quality Assurance Unit
QC	Quality control
RAS	Routine Analytical Services
RCRA	Resource Conservation and Recovery Act
RFA	Resource Conservation and Recovery Act Facility Assessment
RFI	Resource Conservation and Recovery Act Facility Investigation
RPD	Relative percent difference
RPM	Remedial Project Manager
RQAM	Regional Quality Assurance Manager
RRF	Relative Response Factor
RSD	Relative standard deviations
SARMs	Standard analytical reference materials
SAS	Standard Analytical Services
SOP	Standard operating procedure
SOW	Statement of Work
SRM	Standard reference material
SVOC	Semivolatile organic compound
SWMU	Solid waste management unit
TAL	Target analyte list
TCE	Trichloroethene
TCL	Target compound list
TCLEE	Tetrachloroethene
TCLP	Toxicity Characteristic Leaching Procedure
TEPS	Total Environmental Program Support

Glossary

TIC	Tentatively identified compound
TOC	Total organic carbon
TPH	Total petroleum hydrocarbon
TSP	Technical Sampling Plan
USAEC	U.S. Army Environmental Center
USASSC	U.S. Army Soldier Support Center
UTL	Upper tolerance limit
VOC	Volatile organic compound
°C	Degrees Celsius
°F	Degrees Fahrenheit
µg/l	Micrograms per liter
µg/kg	Micrograms per kilogram

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Appendix A

ANALYTICAL LABORATORY STANDARD OPERATING PROCEDURES

UNCONTROLLED COPY**STANDARD
OPERATING
PROCEDURE**

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Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS

SOP No.: Revision No.:
LM-CAL-3001 1.0

Supersedes: Original of December 18, 1989

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Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS

SOP No.: Revision No.:
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1. SCOPE AND APPLICATION

- 1.1 This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices and at part-per-trillion (ppt) concentrations. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits (MCLs) and other germane information. Analysis of a one-tenth aliquot of the sample permits measurement of concentrations up to 10 times the upper MCL (Table 1). Samples containing concentrations of specific congeners (PCDDs and PCDFs) considered within the scope of this method that are greater than the upper MCL must be analyzed by a protocol designed for such concentrations of specific congeners. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is described.
- 1.2 The sensitivity of this method is dependent upon the level of interferences within a given matrix.
- 1.3 This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS).

2. SUMMARY OF METHOD

- 2.1 This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) techniques.
- 2.2 If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination. A simplified analysis flow chart is shown in Figure 1 of the Appendix.

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- 2.3 A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, or fish tissue, is spiked with a solution containing specified amounts of each of the nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Column 1 of Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene (or benzene) Soxhlet extraction for soil, sediment, fly ash samples and aqueous sludges; b) methylene chloride liquid-liquid extraction for water samples; c) dilution of small sample aliquot in hexane for fuel oils and still-bottoms; and d) cyclohexane/methylene chloride extraction for fish tissue; e) ethanol/water extraction for paper pulp. The decision for the selection of an extraction process for chemical reactor residue samples is based on the appearance (consistency, viscosity) of the samples. Generally they can be handled according to the procedure used for still-bottom (or chemical sludge) samples.
- 2.4 The extracts are submitted to an acid-base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding, to the concentrated carbon column eluate, 10 μL (depending on the matrix type) of a tetradecane solution containing 100 $\text{pg}/\mu\text{L}$ of each of the two recovery standards ^{13}C -1,2,3,4-TCDD and ^{13}C -,1,2,3,7,8,9-HxCDD (Table 2). The former is used to determine the percent recoveries of tetra- and pentachlorinated PCDD/PCDF congeners while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF congeners percent recoveries.
- 2.5 One to two μL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

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- 2.6 The identification of OCDD and nine of the fifteen 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C -labeled standard is available in the sample fortification and recovery standard solutions (Table 2), is based on their elution at their exact retention time (-1 to +3 seconds from the respective internal or recovery standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. The remaining six 2,3,7,8-substituted congeners (i.e., 2,3,4,7,8-PeCDF, and 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF, and 1,2,3,4,7,8,9-HpCDF), for which no carbon-labeled internal standards are available in the sample fortification solution, and all other identified PCDD/PCDF congeners are identified by their relative retention times falling within their respective PCDD/PCDF retention time windows, as established by using a GC column performance evaluation solution, and the simultaneous detection of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to ^{13}C -OCDD and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.
- 2.7 Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.
- 2.8 In some instances, samples may be spiked using 1613 standards at 1613 specified levels, and may be quantitated using a 1613 calibration curve.

3. DEFINITIONS

- 3.1 Polychlorinated dibenzo-p-dioxins (PCDDS) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 2) that contain from one to eight chlorine atoms. The fifteen 2,3,7,8-substituted PCDDs (totaling 75) and PCDFs (totaling 135) are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

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- 3.2 Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.
- 3.3 Isomer: Defined by the arrangement of chlorine atoms within an homologous series. For example, 2,3,7,8-TCDD is a TCDD isomer.
- 3.4 Congener: Any isomer of any homologous series.
- 3.5 Internal Standard: An internal standard is a ^{13}C -labeled analog of a congener chosen from the compounds listed in Table 3 and of OCDD. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to measure the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight.
- 3.6 Recovery Standard: Recovery standards (two) are used to determine the percent recoveries for the isotopically labeled PCDDs and PCDFs. The ^{13}C -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated dioxins and furans while ^{13}C -1,2,3,7,8,9-HxCDD permits the recovery determination of the hexa-, hepta- and octachlorinated homologs. They are added to the final sample extract before HRGC/HRMS analysis. Furthermore, ^{13}C -1,2,3,7,8,9-HxCDD is used for the identification of the unlabeled analog present in sample extracts.
- 3.7 High-Resolution Concentration Calibration Solutions (Table 5): Solutions (tetradecane) containing known amounts of 17 selected PCDDs and PCDFs, nine internal standards (^{13}C -labeled PCDDs/PCDFs), and two carbon-labeled recovery standards; the set of five solutions is used to determine the instrument response of the unlabeled analytes relative to the internal standards and of the internal standards relative to the recovery standards.
- 3.8 Sample Fortification Solution (Table 2): A solution (isooctane or toluene) containing the nine internal standards, which is used to spike all samples before extraction and cleanup.

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- 3.9 Recovery Standard Solution (Table 2): A tetradecane solution containing the two recovery standards, which is added to the final sample extract before HRGC/HRMS analysis.
- 3.10 Field Blank: A portion of a sample representative of the matrix under consideration, which is free of any PCDDs/PCDFs.
- 3.11 Laboratory Method Blank: A blank prepared in the laboratory and carried through all analytical procedure steps except the addition of a sample aliquot to the extraction vessel.
- 3.12 Rinsate: A portion of solvent used to rinse sampling equipment. The rinsate is analyzed to demonstrate that samples were not contaminated during sampling.
- 3.13 GC Column Performance Check Mixture: A tetradecane solution containing a mixture of selected PCDD/PCDF standards including the first and last eluters for each homologous series, which is used to demonstrate continued acceptable performance of the capillary column (i.e., ≤ 25 percent valley separation of 2,3,7,8-TCDD from all the other 21 TCDD isomers) and to define the homologous PCDD/PCDF retention time windows.
- 3.14 Performance Evaluation Materials: Representative sample portions containing known amounts of certain unlabeled PCDD/PCDF congeners (in particular the ones having a 2,3,7,8-substitution pattern). Representative interferences may be present. PEMs are obtained from the EPA EMSL-LV and submitted to potential contract laboratories, must analyze these and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). PEMs are also included as unspecified ("blind") quality control (QC) samples in any sample batch submitted to a laboratory for analysis.
- 3.15 Relative Response Factor: Response of the mass spectrometer to a known amount of a native analyte relative to a known amount of an internal standard, or a known amount of internal standard to a known amount of a recovery standard.

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- 3.16 Estimated Level of Method Blank Contamination: The response from a signal occurring in the homologous PCDD/PCDF retention time windows, at any of the masses monitored, is used to calculate the level of contamination in the method blank. The results from such calculations must be reported along with the data obtained on the samples belonging to the batch associated with the method blank.
- 3.17 Batch: A group of samples processed at the same time.
- 3.18 Sample Rerun: Extraction of another portion of the sample followed by extract cleanup and extract analysis.
- 3.19 Extract Reanalysis: Analysis by HRGC/HRMS of another aliquot of the final extract.
- 3.20 Mass Resolution Check: Standard method used to demonstrate a static resolving power of 10,000 minimum (10 percent valley definition).
- 3.21 Method Calibration Limits (MCLs): For a given sample size, a final extract volume, and the lowest and highest concentration calibration solutions, the lower and upper MCLs delineate the region of quantification for which the HRGC/HRMS system was calibrated with standard.
- 3.22 Matrix Spike (MS): A sample which is spiked with a known amount of the matrix spike fortification solution (this exhibit, Section 3.24) prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.
- 3.23 Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.
- 3.24 Matrix Spike Fortification Solution: Solution used to prepare the MS and MSD samples. It contains all unlabeled analytes listed in Table 5. The solution also contains all internal standards used in the sample fortification solution per the method.

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4. COMMENTS

- 4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts should avoid using PVC gloves.
- 4.2 The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.
- 4.3 Reuse of glassware is to be minimized to avoid the risk of contamination.
- 4.4 Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.
- 4.5 A high-resolution capillary column (60 m DB-5) is used to resolve as many PCDD and PCDF isomers as possible; however, no single column is known to resolve all isomers. The use of several capillary columns will, in fact, be necessary during the determination of the toxicity equivalency factors (TEFs) this exhibit, Section 14.7).

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5. SAFETY ISSUES

- 5.1 2,3,7,8-TCDD is identified as a carcinogen, teratogen, and mutagen. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD.
- 5.2 The analyst should note that finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box. Laboratory personnel handling these types of samples should also wear masks fitted with charcoal filter absorbent media to prevent inhalation of dust.
- 5.3 Safety practices described in Sections 5.4 through 5.5 are adapted from EPA Method 613, Section 4 (July 1982 version).
- 5.4 The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should also be made available to all personnel involved in the chemical analysis. Personnel are expected to read pertinent MSDS's before handling chemicals or samples.
- 5.5 Each laboratory must develop a strict safety program for the handling of 2,3,7,8-TCDD. The laboratory practices listed below are recommended.
 - 5.5.1 Contamination of the laboratory will be minimized by conducting the manipulations in a fume hood.

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- 5.5.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.
- 5.5.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light at a wavelength less than 290 nm for several days (use F 40 BL lamps or equivalent). Using this analytical method, analyze the liquid wastes and dispose of the solutions when 2,3,7,8-TCDD can no longer be detected.
- 5.6 Some of the following precautions were issued by Dow Chemical U.S.A. (revised 11/78) for safe handling of 2,3,7,8-TCDD in the laboratory and amended for use in conjunction with this method.
 - 5.6.1 The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. The 2,3,7,8-TCDD isomer is extremely toxic to certain kinds of laboratory animals. However, it has been handled for years without injury in analytical laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.
 - 5.6.1.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and laboratory hood adequate for radioactive work.
 - 5.6.1.2 Training: Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

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- 5.6.1.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).
- 5.6.1.4 Confinement: Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.
- 5.6.1.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.
- 5.6.1.6 Disposal of Hazardous Wastes: Refer to the November 7, 1986 issue of the Federal Register on Land Ban Rulings for details concerning the handling of dioxin-containing wastes.
- 5.6.1.7 Decontamination: Personnel - any mild soap with plenty of scrubbing action. Glassware, tools and surfaces - Chlorothene NU solvent (Trademark of the Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished rinsing with Chlorothene, then washing with any detergent and water. Dish water may be disposed to the sewer after percolation through a charcoal bed filter. It is prudent to minimize solvent wastes because they require special disposal through commercial sources that are expensive.
- 5.6.1.8 Laundry: Clothing known to be contaminated should be disposed with the precautions prescribed under "Disposal of Hazardous Wastes." Laboratory coats or other clothing worn in 2,3,7,8-TCDD work area may be laundered. Clothing should be collected in plastic bags.

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5.6.1.9 Wipe Tests: A useful method of determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract. (See Appendix)

5.6.1.10 Inhalation: Any procedure that may produce airborne contamination must be carried out with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no significant inhalation hazards except in case of an accident.

5.6.1.11 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

6. APPARATUS

6.1 High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.1.1 The GC must be equipped for temperature programming, and all required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On-Column 1-uL injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2-uL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 uL). One-uL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times.

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- 6.1.2 Gas Chromatograph/Mass Spectrometer (GC/MS) Interface--The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel(TM) or equivalent ferrules are recommended.
- 6.1.3 Mass Spectrometer--The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less (this exhibit, Section 9.1.4.1). At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes prior to elution of the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ¹³C-HxCDF and ¹³C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The selection of the lock-mass ion is left to the performing laboratory. The recommended mass spectrometer tuning conditions (this exhibit, Section 8.2.3) are based on the groups of monitored ions shown in Table 6.

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6.1.4 Data System -- A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data at a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles (this exhibit, Section 8.2.4) and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.2 GC Column

In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. Minimum acceptance criteria must be demonstrated and documented (this exhibit, Section 8.1). At the beginning of each 12-hour period (after mass resolution is demonstrated) during which sample extracts or concentration calibration solutions will be analyzed, column operating conditions must be attained for the required separation on the column to be used for samples. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

6.3 Miscellaneous Equipment and Materials

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

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- 6.3.1 Nitrogen evaporation apparatus with variable flow rate.
- 6.3.2 Balances capable of accurately weighing to 0.01 g and 0.0001 g.
- 6.3.3 Centrifuge.
- 6.3.4 Water bath, equipped with concentric ring covers and capable of maintaining temperature control within +/- 2° C.
- 6.3.5 Stainless steel or glass containers large enough to hold contents of one-pint sample containers.
- 6.3.6 Glove box.
- 6.3.7 Drying oven.
- 6.3.8 Stainless steel spoons and spatulas.
- 6.3.9 Laboratory hoods.
- 6.3.10 Pipets, disposable, Pasteur, 150 mm long x 5 mm ID.
- 6.3.11 Pipets, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.2.
- 6.3.12 Reacti-vial, 2 mL, silanized amber glass.
- 6.3.13 Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.
- 6.3.14 Separatory funnels, 125 mL.
- 6.3.15 Kuderna-Danish concentrator, 500 mL, fitted with 10-mL concentrator tube and three-ball Snyder column.
- 6.3.16 Teflon(TM) boiling chips (or equivalent), washed with DCM before use.

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- 6.3.17 Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon(TM) stopcock.
- 6.3.18 Adaptors for concentrator tubes.
- 6.3.19 Glass fiber filters.
- 6.3.20 Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125-mL flask.
- 6.3.21 Continuous liquid-liquid extractor.
- 6.3.22 All-glass Soxhlet apparatus, 500-mL flask.
- 6.3.23 Glass funnels, sized to hold 170 mL of liquid.
- 6.3.24 Desiccator.
- 6.3.25 Solvent reservoir (125 mL), Kontes; 12.35 cm diameter (special order item), compatible with gravity carbon column.
- 6.3.26 Rotary evaporator with a temperature-controlled water bath.
- 6.3.27 High-speed tissue homogenizer, equipped with an EN-8 probe or equivalent.
- 6.3.28 Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

NOTE: Reuse of glassware should be minimized to avoid the risk of contamination. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure:

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6.4 Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by absorption on the glassware surface.

6.4.1 Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

6.4.2 After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

6.4.3 Do not bake reusable glassware in an oven as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated baking of glassware may cause the formation of active sites on the glass surface that will irreversibly absorb PCDDs/ PCDFs.

6.4.4 Immediately prior to use, Soxhlet extraction glassware should be pre-extracted with toluene for approximately 3 hours.

7. REAGENTS AND STANDARDS

7.1 Column Chromatography Reagents

7.1.1 Silica Gel - Kieselgel 60 or equivalent, activate for >12 hours at 130°C before use. Store at 130°C in covered flask.

7.1.2 Acid Alumina - Bio-Rad Ag-4 or equivalent, activate for >12 hours at 130°C before use. Store at 130°C in covered flask.

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- 7.1.3 Basic Alumina - Bio-Rad Ag-10 or equivalent, kiln at 600°C for >24 hours before use. Store at 130°C in covered flask. DO NOT USE IF OLDER THAN 5 DAYS!
- 7.1.4 Carbopack/silica gel - Mix 3.6g carbopack (Supelco 1-0257) and 16.4 g activated silica gel; (alternatively, prepare AX-21/silica gel (5%/95%); i.e., combine 5 g precleaned AX-21 with 95 g silica gel). Activate mix for >12 hours at 130°C before use. Store at 130°C in covered flask.
- 7.1.5 44% H₂SO₄/silica gel - Mix 24mL conc. H₂SO₄ and 56g activated silica gel. Stir and shake until free flowing. Store at room temperature.
- 7.1.6 33% NaOH/silica gel - Mix 34mL 1N NaOH and 67g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2 Reagents

- 7.2.1 Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.
- 7.2.2 Potassium hydroxide, ACS grade, 20 percent (w/v) in distilled water.
- 7.2.3 Distilled water demonstrated to be free of interferents
- 7.2.4 Potassium carbonate, anhydrous, analytical reagent.
- 7.2.5 Silica gel.

7.3 Desiccating Agent

- 7.3.1 Sodium sulfate, granular, anhydrous; use as such.

7.4 Solvents

- 7.4.1 High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, benzene, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

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7.5 Calibration Solutions

7.5.1 High-Resolution Concentration Calibration Solutions (Table 5) -- Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 11) PCDDs and PCDFs at known concentrations used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (1.0 pg/uL) and the highest for the octachlorinated congeners (1000 pg/uL).

7.5.2 Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.5.3 Store the calibration solutions in capped test tubes and at room temperature in the dark.

7.6 GC Column Performance Check Solution

This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ¹³C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/uL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution.

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7.7 Sample Fortification Solution (Matrix Spike Mix)

This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C -OCDF is not present in the solution.)

7.8 Recovery Standard Solution

This tetradecane solution contains two recovery standards (^{13}C -1,2,3,4-TCDD and ^{13}C -1,2,3,7,8,HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SYSTEM PERFORMANCE CRITERIA

System performance criteria are presented below. The laboratory may use the recommended GC column described in Section 6.2. It must be documented that all applicable system performance criteria specified in Section 8.1 were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 4 provides a typical 12-hour analysis sequence. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

8.1 GC Column Performance

- 8.1.1 Inject 2 μL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of ≤ 1 second.

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- 8.1.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of ≤ 25 percent (Figure 5), where

$$\text{Valley Percent} = (x/y) (100)$$

x = measured as in Figure 5 from the 2,3,7,8-closest TCDD eluting isomer, and

y = the peak height of 2,3,7,8-TCDD.

It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

- 8.1.3 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

8.2 Mass Spectrometer Performance

- 8.2.1 The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

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8.2.2 Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

8.2.3 By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using the peak matching unit and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

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8.2.4 Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low-mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 6) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

9. CALIBRATION

9.1 Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (this exhibit, Section 9.3) does not meet the required criteria listed in Section 9.4 (this exhibit).

9.1.1 Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

9.1.2 Tune the instrument with PFK as described in Section 8.2.3.

9.1.3 Inject 2 μ L of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 8.1. The total cycle time must be ≤ 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criterion listed in Section 8.1.2 is met.

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- 9.1.4 By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 2- μ L portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.
- 9.1.4.1 The total cycle time for data acquisition must be 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.
- 9.1.4.2 Acquire SIM data for all the ions listed in the five descriptors of Table 6.
- 9.1.4.3 The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).
- 9.1.4.4 The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards must be within the control limits stipulated in Table 9.
- NOTE: Sections 9.1.4.3 and 9.1.4.4 require that 17 ion ratios from Section 9.1.4.3 and 11 ion ratios from Section 9.1.4.4 be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.
- 9.1.4.5 For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

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9.1.4.6 Referring to Table 10, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate internal standards (Table 5) and the nine RRFs for the labeled ¹³C internal standards [RRF(m); m=18 to 26] relative to the two recovery standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

$$RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled recovery standards,

Q_{is} = quantity of the internal standard injected (pg),

Q_{rs} = quantity of the recovery standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF(n) and RRF(m) are dimensionless quantities; the units used to express Q_{is} , Q_{rs} , and Q_x must be the same.

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- 9.1.4.7 Calculate the $\overline{RRF}(n)$ s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = 1/5 \sum_{j=1}^5 RRF_j(n)$$

where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 10), and j is the injection number (or calibration solution number; j = 1 to 5).

- 9.1.4.8 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 10) are calculated as follows:

- 9.1.4.8.1 For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 9.1.4.7.

NOTE: The calibration solutions do not contain ^{13}C -OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of ^{13}C -OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from ^{13}C -OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C -OCDD.

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9.1.4.8.2 For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{\text{RRF}}(k) = \frac{1}{t} \sum_{n=1}^t \text{RRF}_n$$

where

k = 27 to 30 (Table 10), with 27 = PeCDF; 28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

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9.1.4.9 Relative response factors [$\overline{RRF}(m)$] to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RRF(m) = \frac{A_{is}^m \times Q_{rs}}{Q_{is}^m \times A_{rs}}$$

$$\overline{RRF}(m) = \frac{1}{5} \sum_{j=1}^5 RRF_j(m),$$

where:

m = 18 to 26 (congener type) and $j = 1$ to 5 (injection number),

A_{is}^m = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard ($m = 18$ to 26),

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard ($m = 18$ to 26),

Q_{rs} & Q_{is}^m = quantities of, respectively, the recovery standard (rs) and a particular internal standard (m) injected (pg),

$RRF(m)$ = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and

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$\overline{\text{RRF}}(\text{m})$ = calculated mean relative response factor of a particular internal standard, as determined from the five initial calibration injections (j).

9.2 Criteria For Acceptable Calibration

The criteria listed below for acceptable calibration must be met before the analysis is performed.

9.2.1 The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must not exceed ± 20 percent, and those for the nine labeled reference compounds must not exceed ± 30 percent.

9.2.2 The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10 .

9.2.3 The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 9.2.1 (this exhibit) is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (this exhibit, Section 9.4) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

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9.3 Routine Calibration (Continuing Calibration Check)

Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks.

9.3.1 Inject 2 uL of the concentration calibration solution HRCC-3 containing 10 pg/uL of tetra- and pentachlorinated congeners, 25 pg/uL of hexa- and heptachlorinated congeners, 50 pg/uL of octachlorinated congeners, and the respective internal and recovery standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 and 6.2 (this exhibit), determine and document an acceptable calibration as provided in Section 9.4 (this exhibit).

9.4 Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken.

- 9.4.1 The measured RRFs [RRF(n) for the unlabeled standards] obtained during the routine calibration runs must be within 20 percent of the mean values established during the initial calibration (this exhibit, Section 9.1.4.7).
- 9.4.2 The measured RRFs [RRF(m) for the labeled standards] obtained during the routine calibration runs must be within 30 percent of the mean values established during the initial calibration (this exhibit, Section 9.1.4.9).
- 9.4.3 The ion-abundance ratios (Table 9) must be within the allowed control limits.
- 9.4.4 If either one of the above criteria (this exhibit, Sections 9.4.1 and 9.4.2) is not satisfied, the entire initial calibration process (this exhibit, Section 9.1) must be repeated. If the ion-abundance ratio criterion (this exhibit, Section 9.4.3) is not satisfied, refer to the note in Section 9.1.4.4 (this exhibit) for resolution.

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NOTE: An initial calibration must be carried out whenever the HRCC-3, the sample fortification or the recovery standard solution is replaced by a new solution from a different lot.

10. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, HOLDING TIMES, TREATMENTS, AND ANCILLARY DETERMINATIONS

10.1 The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

10.2 Grab and composite samples must be collected in glass containers.

Conventional sampling practices must be followed. The bottle must not be prewashed with sample before collection. Sampling equipment must be free of potential sources of contamination.

10.3 Grinding or Blending of Fish Samples.

If not otherwise specified by the EPA, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meatgrinder with a 3- to 5-mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the EPA. If so requested by the EPA, the above whole fish requirement is superseded.

10.4 With the exception of the fish tissues, which must be stored at -20°C, all samples must be stored at 4°C, extracted within 30 days and completely analyzed within 45 days of collection.

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10.5 Phase Separation

10.5.1 On a routine basis, very wet soil and sediments may be air dried prior to percent moisture determination and extraction procedures.

10.5.2 Non-routinely, phase separation on very wet (>25 percent water) soil and sediment samples may be accomplished as follows. Place a 50-g portion in a suitable centrifuge bottle and centrifuge for 30 minutes at 2,000 rpm. Remove the bottle and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipet, transfer the liquid layer into a clean bottle. Mix the solid with a stainless steel spatula and remove a portion to be weighed and analyzed (percent moisture determination, extraction). Return the remaining solid portion to the original sample bottle (empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil and sediment method. Take note of and report the estimated volume of liquid before disposing of the liquid as a liquid waste.

CAUTION: Finely divided soils and sediments contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing PCDDs/PCDFs (including 2,3,7,8-TCDD). Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

10.6 Soil, Sediment or Paper Sludge (Pulp) Percent Moisture Determination.

The percent moisture of soil or sediment samples showing detectable levels (see note below) of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined according to the following recommended procedure.

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Generally, depending on sample availability, a 5-10 g sample, weighed to three significant figures, is used for % solids determination. The sample is then dried to constant weight at 100°C in an adequately ventilated oven. Weigh the dried solid to three significant figures. Calculate and report the percent moisture on the appropriate form. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

NOTE: The lower MCLs (Table 1) may be used to estimate the minimum detectable levels.

$$\text{Percent Moisture} = \frac{\text{Weight of wet soil} - \text{Weight of dry soil}}{\text{Weight of wet soil}} \times 100$$

10.7 Fish Tissue Lipid Content Determination

The percent lipid of fish samples showing detectable levels of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined as follows:

Use a separate portion (2 g) of the ground frozen fish sample. Blend it with 6 g anhydrous sodium sulfate, pour the mixture into a 1-cm i.d. glass column and extract the lipids by passing two 25-mL portions of methylene chloride through the column and collecting the extract in a tared 100-mL round-bottom flask. Concentrate the extract on a rotary evaporator until constant weight is attained. The percent lipid is calculated using the following expression:

$$\text{Percent lipid} = \frac{\text{Weight of residue from extraction (in g)}}{\text{Weight of fish tissue portion (in g)}} \times 100$$

Dispose of the lipid residue as a hazardous waste if the results of the analysis indicate the presence of PCDDs or PCDFS.

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11. EXTRACTION AND CLEANUP PROCEDURES

11.1 Internal Standard Addition. Use a portion of 1 g to 1000 g (typical sample size requirements for each type of matrix are given in Section 11.2 of this exhibit and in Table 1) of the sample to be analyzed. Transfer the sample portion to a tared flask and determine its weight. Add an appropriate quantity of the sample fortification mixture to the sample. A 100 uL aliquot of fortification mixture is added to all samples, regardless of sample size. As an example, for ^{13}C -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 1000 pg of ^{13}C -2,3,7,8-TCDD to give the requisite 100 ppt fortification level.

11.2 Extraction

11.2.1 Sludge - Paper Pulp Sludges are generally air-dried and ground. Because of the drying procedure, a Dean-Stark water separator may, or may not, be used for extraction. Extraction is generally done by Soxhlet with 200-300 mL of Ethanol/Toluene 68:32.

Non-Paper Pulp Sludges are extracted with 200-300 mL of toluene.

Soxhlet sample for a minimum of 16 hrs. Cool the sample, filter the toluene (or benzene) extract, if needed, through a glass-fiber filter, or equivalent, into a round-bottom flask. Rinse the filter with 10 mL toluene (or benzene), and combine the extract and rinsate. Concentrate the combined solutions to near dryness on a rotary evaporator at 50°C (toluene) or a Kuderna-Danish (KD) apparatus (benzene). Use of an inert gas to concentrate the extract is also permitted. Proceed with Section 11.2.4. below.

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- 11.2.2 Still-Bottom/Fuel Oil. All organic liquids and solids that will dissolve in a solvent will be treated as a solvent dilution. Dissolve 1-2mL of sample in an appropriate solvent; then dilute with 125mL of Hexane. Spike with appropriate Internal Standards and proceed with section 11.3
- 11.2.3 Fly Ash. Extract fly ash samples by placing a sample portion (e.g., 10 g) and an equivalent amount of anhydrous sodium sulfate in a Soxhlet extraction apparatus charged with 200-300 mL toluene (or benzene), and extract for 16 hours using a three cycle/hour schedule. Cool and filter the toluene (or benzene) extract through a glass-fiber filter into a 500-mL round-bottom flask. Rinse the filter with 5 mL toluene (or benzene). Concentrate the combined toluene (or benzene) solutions to near dryness on a rotary evaporator (toluene) at 50°C or a KD apparatus (benzene). Proceed with Section 11.2.5.4 below.
- 11.2.4 Soil. Add anhydrous sodium sulfate to the soil sample portion in a ratio of 2 to 1 (e.g. 20g sodium sulfate to 10g of sample) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass-wool plug (the use of an extraction thimble is optional). Add 200 to 250 mL benzene (or toluene) to the Soxhlet apparatus and reflux for 16 hours. The solvent must cycle completely through the system at least three times per hour. Proceed with Section 11.2.5.4.
- 11.2.5 Aqueous Samples. Mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Pour the entire sample (approximately 1-L) into a 2-L separatory funnel. Proceed with Section 11.2.5.1 (this exhibit).

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NOTE: A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel. Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the extractor. Repeat the sample bottle rinse with an additional 50- to 100-mL portion of methylene chloride and add the rinsate to the extractor. Add 200 to 500 mL methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 hours. Allow to cool, then detach the distilling flask. Proceed with Section 11.2.5.3.

11.2.5.1 Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. Extraction is repeated two additional times with DCM.

11.2.5.2 Determine the original sample volume by filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11.2.5.3 Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with DCM and load funnel with DCM-rinsed Na₂SO₄. Pour extract through Na₂SO₄ to remove water. Rinse Na₂SO₄ with fresh DCM and collect in round bottom flask.

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11.2.5.4 Transfer the extract to a 500-mL round-bottom, add 100 μ l of tetradecane and concentrate on a rotary evaporator or TurboVap.

- 11.3 Partition the extract in 50-125 mL of hexane against 40 mL concentrated sulfuric acid in a separatory funnel. Shake for two minutes. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).
- 11.4 Partition the extract against 50 mL distilled H₂O (w/v). Shake for two minutes. Remove and discard the aqueous layer (bottom).
- 11.5 Partition the extract using 50 mL of 10 Normal Sodium Hydroxide. Shake for two minutes. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base is known to degrade certain PCDDs/PCDFs, so contact time must be minimized.
- 11.6 Partition the extract against 50 mL of distilled H₂O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15-mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.)
- 11.7 Proceed with the following Clean-up Steps:
- For NON paper pulp mill solids & effluents proceed with clean-up step exhibit "A".
- For paper pulp mill solids & effluents proceed with exhibit "B".

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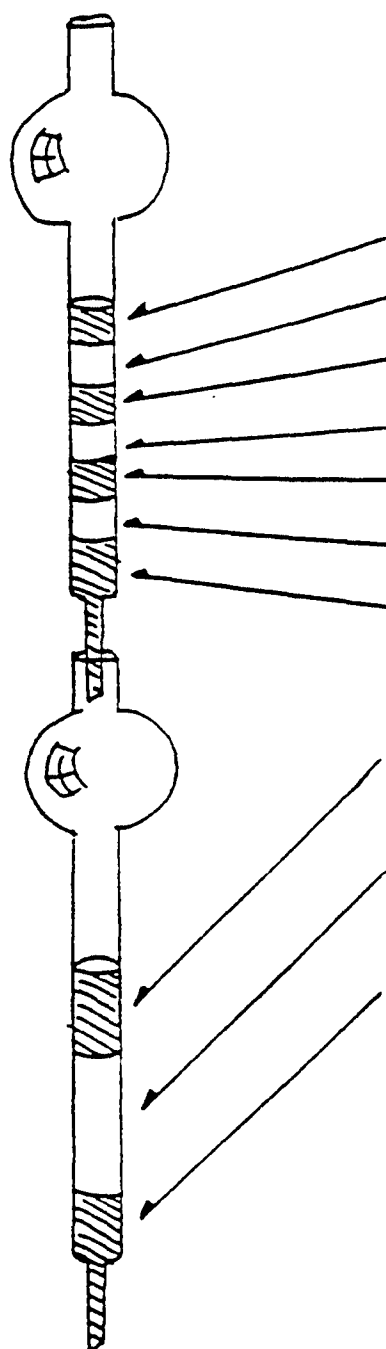
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EXHIBIT A

IFB COLUMN CLEANUP

Use 15 mm column for top column
 Use 11 mm column for bottom column



	# of 15mL capsfull
1 cm Na ₂ SO ₄	1
2g Silica gel	2
4g 44% H ₂ SO ₄ /Silica gel	2
1g Silica gel	1
2g 33% 1M NaOH/silica gel	1
1g Silica gel	1
glasswool	

1 cm Na₂SO₄
 8g Acid alumina
 glasswool

- Pre-rinse both columns with hexane 40 mL Top and 20 mL Bottom
- Put one column above the other
- Add extract to the top column - Rinse Extract Vessel 2 times with 1 mL ea. of Hexane and add to column.
- Elute the top column directly onto the bottom column with 90mL hexane
- Elute the bottom column with 20 mL of hexane - discard in proper waste stream
- Elute with 20mL of 20% MeCl₂/hexane
- Collect eluate in Turbo vap tube
- Use tetradecane and N₂ as appropriate per flow chart

Proceed with 11.8

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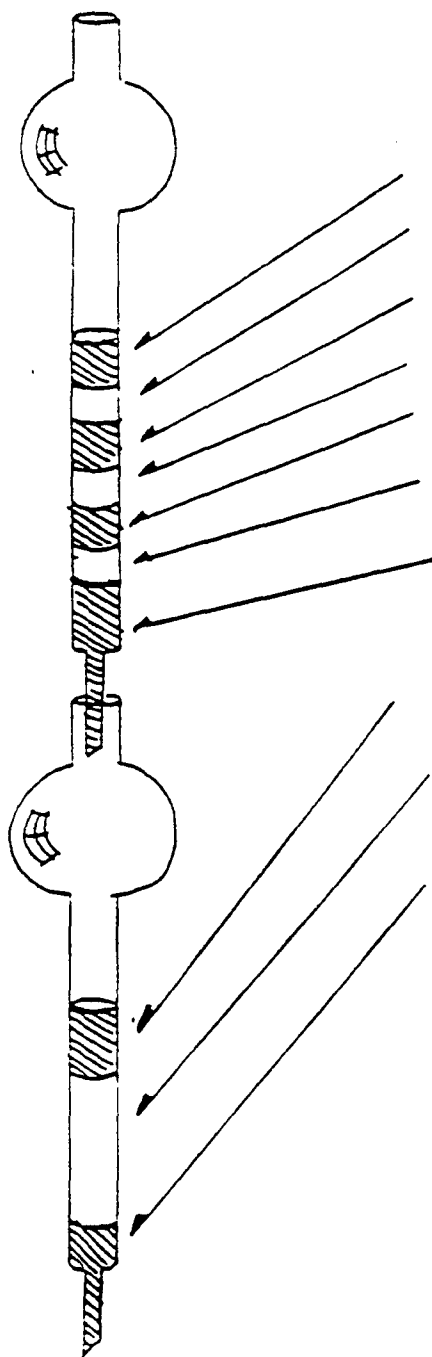
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EXHIBIT B

NCASI Paper Pulp IFB COLUMN CLEANUP

Use 15 mm column for top column
 Use 11 mm column for bottom column



	# of 15mL capsfull
1 cm Na ₂ SO ₄	1
2g Silica gel	2
8g 44% H ₂ SO ₄ /Silica gel	4
1g Silica gel	1
4g 33% 1M NaOH/silica gel	2
1g Silica gel	1
glasswool	

1 cm Na₂SO₄
 8g Acid alumina
 glasswool

- Pre-rinse both columns with hexane 40 mL Top and 20 mL Bottom
- Put one column above the other
- Add extract to the top column w/hexane - Rinse Extract Vessel 2 times with 1 mL ea. of Hexane and add to column
- Elute the top column directly onto the bottom column with 120mL hexane (60 x 2)
- Elute the bottom column with 20 mL of hexane - discard in proper waste stream
- Elute with 23mL of 20% MeCl₂/hexane
- Collect eluate in Turbo vap tube
- Use tetradecane and N₂ as appropriate per flow chart

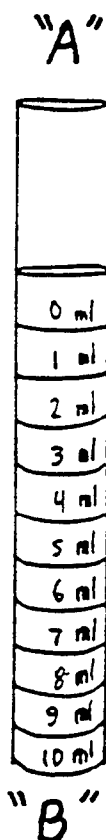
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- 11.8 Carbon Column Cleanup - Prepare an AX-21 Carbon & Silica Gel column as described in EXHIBIT D, Page 41.



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EXHIBIT D
CARBON COLUMN CLEANUP
SPECIAL D2

- Cut off both ends of a 10 mL pipet.
- Push a glasswool plug down to the 6 mL mark.
- Add 1g of 5% AX-21/silica and top with another glasswool plug.

- Pre-elute with 5 mL 1:1 MeCl₂:cyclohexane. Direction "A"
- Turn over and pre-elute with 5 mL 1:1 MeCl:cyclohexane in direction "B"..
- Discard pre-eluates.

- Dilute extract to 1 mL with hexane and transfer to the column.
- Rinse sample vial onto the column with 2 x 2 mL 1:1 MeCl₂:cyclohexane

- Elute with: 6mL 1:1 MeCl₂:cyclohexane
5mL 75:20:5 MeCl₂:MeOH:Benzene

- DISCARD ELUATES

- Turn the column over and elute with 25 mL toluene in direction "A".

- N₂ or roto-vap to NEAR dryness and proceed to next step.

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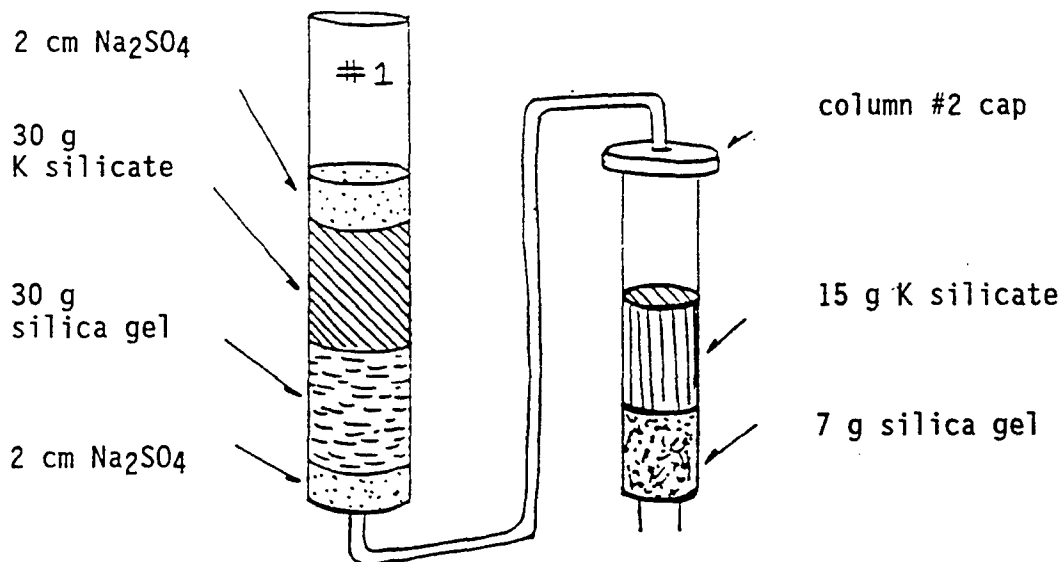
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11.9 Extraction and Purification Procedures for Biota (Stallings SOP)

Column #1 & #2 - Use approximately 850 mL of Solvent A (1:1 cyclohexane:DCM) per sample: 700 mL through the column, 100 mL into the jar with the sample before spiking, and 50 mL to rinse the jar.

NOTE: (Add 30 g anhydrous sodium sulfate to a 10-g portion of a homogeneous tissue sample and mix thoroughly with a stainless steel spatula. After breaking up any lumps, prepare to add the tissue/sodium sulfate mixture to the top of column). Use a 2 mL pipet to stir up the sample. Then pour the spiked sample through a large funnel onto the #1 dry column, using 50mL to rinse the sample jar. Keep the small column wet with Solvent A. When the solvent reaches the top cap of the small column (#2), fill the small column to the top and place the cap on tightly. When the Solvent A reaches the top of the solid material in the column, pour 700 mL of solvent A (in two portions) onto the column. Drain into a 1 L clear glass jar (CGJ).



Column #1: Flex Column number 420400-2550
Column #2: Flex Column number 420400-1530

Disposal of columns: Air out the columns in a hood. Take the used columns #1 & #2 and place them in a doubled garbage bag. Seal with red tape and dispose of in the lab pack.

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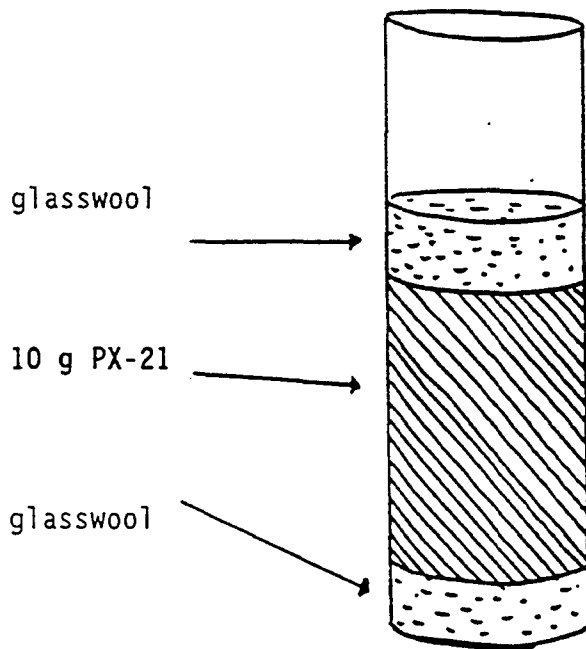
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Column #3 - Close the stopcock. Put glasswool in the bottom of the column. Weigh 10 g of AX-21/silica gel in a 150 mL beaker, add 75 mL of toluene, mix, and pour into column #3. Rinse beaker with toluene. After the packing settles, drain the toluene down to the top of the packing material. Rinse the sides of the column with MeOH and drain down to 2 cm above the top of the packing material. Put a plug of glasswool on top of the column. Condition the column with 80 mL of MeOH, discard the eluant. Then elute the column with 80 mL of Solvent A and discard.

Add Cleanup Recovery Standard (CRS) to the samples. Then add entire extract from column 2 to the top of column 3. Drain samples through the column and discard the eluate. Drain 75 mL of Solvent A through the column, discard. Drain 50 mL 75:20 DCM:MeOH through the column, discard.

Turn the column over and put a filter funnel and 250 mL round bottom flask containing 100 μ L C, under the column. Insert the reservoir column and fill with 75 mL of Toluene. Elute the packed column, collecting the toluene in the 250 mL round bottom flask. Carefully blow out the toluene in the packing material with a squeeze bulb.



Proceed with columns #4 and #5

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Column #4 & #5 - Use a 5 mL pipet with a glasswool plug to estimate the acid alumina for column #5. Place the plug at the 4.5 mL mark and measure up to the Zero mark, making sure to pack the material by tapping on the pipet. Once you have 4.5 mL of acid alumina, pour into column #5. Add a little over 1/4 inch of DCM rinsed Na₂SO₄ and place the cap back on the column. Insert a pasteur pipet containing a small glass woolplug (column #4) into the small hole in the cap of column #5. Add about 1 cm of acid silica into column #4. Add about 1 cm K-silicate on top of the acid silica.

Set up test tube rack, raised with a 1 inch board, with 8 mL test tubes and 40 mL VOA vials. The first 40 mL VOA vial is for the 23 mL of pre-rinse, the 8 mL test tube is for the post-rinse, and the 2nd 40 mL VOA vial is for the 37 mL of remaining eluate. Label the containers with the appropriate CAL ID#.

At this point, sample extracts from column 3 should be roto-vap'd down to the tetradecane and brought up to 5 mL in hexane. Set up samples (250 mL round bottom) in front of the columns.

Solvents: 2% DCM:Hexane
 5% DCM:Hexane
 8% DCM:Hexane
 Hexane

Place first 40 mL VOA vial underneath each of the column #5's. Transfer all of sample to column #4. Fill column #4 to the top with hexane. Add another 5 mL to the sample round bottom. After the solvent in column #4 reaches the Na₂SO₄, transfer the 5 mL in the round bottom onto the column. Blow out column #4 using a red pipette bulb (discard column #4). Rinse the reusable reservoir (yellow top) with hexane and insert it into the top of column #5. Add 10 mL of 2% solvent. Transfer the labels from the round bottom to the second 40 mL VOA vial.

The first VOA vial should contain two 5 mL hexane rinses and the 10 mL of 2% solvent. Cap and archive the rinses.

Place the second 40 mL VOA vial under the columns; eluate and collect:
 Add 5 mL 2% solvent
 Add 15 mL 5% solvent
 Add 20 mL 8% solvent

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Remove the VOA vial. Place a labeled 8 mL test tube under the column #5 and elute with 5 mL of DCM. Archive as a post rinse. Label test tube with the appropriate CAL ID#.

Reduce the volume of the second VOA vial with N₂ to a small puddle. Transfer the sample to a 2 mL conical vial including 0.5 mL rinse of the VOA vial. Do not blow the sample to dryness in the 40 mL VOA vial.

Column #4: Pasteur pipet
Column #5: Flex Column number 420400-0730

Proceed with 12.1

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12. ANALYTICAL PROCEDURES

- 12.1 With a stream of dry, purified nitrogen, reduce the extract volume to 10 uL. Add 10 uL of the recovery standard solution (Table 2).
- 12.2 Inject a 1 or 2uL aliquot of the extract into the GC, operated under the conditions previously used (this exhibit, Section 6.2) to produce acceptable results with the performance check solution.

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12.3 Acquire SIM data according to Section 6.1.3 (this exhibit). Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (this exhibit, Sections 9.1.4.6 through 9.1.4.9). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is to monitor their interference during the characterization of PCDFs.

12.4 Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.4.1 Retention Times.

12.4.1.1 For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Tables 2 and 3), the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled internal or recovery standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.4.1.2 For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract (this represents a total of six congeners), the relative retention time (relative to the appropriate internal standard) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C -OCDD as determined from the daily routine calibration results.

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12.4.1.3 For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.4.1.4 The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8465 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.4.1.5 The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C -TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.4.2 Ion Abundance Ratios

12.4.2.1 The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Sections 9.1.4.3 and 9.1.4.4 (this exhibit) and Table 9 for details.

12.4.3 Signal-To-Noise Ratio

12.4.3.1 All ion current intensities must be ≥ 2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 7 describes the procedure to be followed for the determination of the S/N.

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12.4.4 Polychlorinated Diphenyl Ether Interferences

12.4.4.1 In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N ≥ 2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

13. QA/QC REQUIREMENTS

13.1 Performance Check Solutions

13.1.1 At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 3 (HRCC-3) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed.

13.1.2 Deviations from criteria specified for the GC performance check or for the mass resolution check check invalidates all positive sample data collected between analysis of the performance check solution; extracts from those positive samples shall be reanalyzed.

If the routine calibration run fails at the beginning of a 12-hour shift, the instructions in Section 9.4.4 must be followed.

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13.1.3 The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted PCDDs/PCDFs, which are not obtained from the EMSL-CIN, must be verified by comparison with the EPA standard solutions that are available from the EMSL-CIN.

13.2 Blanks

13.2.1 Method Blank (MB)

One method blank is required per batch or per 20 samples, whichever is more frequent. For the method blank use all reagents, standards, equipment, apparatus, glassware and solvents that would be used for a sample. If the accompanying samples are aqueous, use methylene chloride-rinsed distilled water as a matrix. (There is no matrix for the MB if samples are nonaqueous.) Take the MB through all steps detailed in the analytical procedure.

13.2.1.1 The method blank must contain the same amount of ^{13}C -labeled internal standards that is added to samples before extraction.

13.2.1.1.1 If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

13.2.1.1.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

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13.2.2 Field Blanks

Each batch of samples may contain a field blank sample of uncontaminated soil, sediment or water that is to be fortified before analysis.

13.2.2.1 Fortified Field Blank

13.2.2.1.1 Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add 100 μ L of the solution containing the nine internal standards (Table 2) diluted with 1.5 mL acetone.

13.2.2.1.2 Extract by using the procedures described in Section 11.2. As applicable, add 10 μ L of the recovery standard solution and analyze a 2- μ L aliquot of the concentrated extract.

13.2.2.2 Calculate the concentration of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards.

13.2.2.2.1 Extract and analyze a new simulated fortified field blank whenever new lots of solvents or reagents are used for sample extraction or for column chromatographic procedures.

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13.2.2.3 Rinsate Samples

- 13.2.2.3.1 In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.
- 13.2.2.3.2 The rinsate sample must be fortified like a regular sample.
- 13.2.2.3.3 Take a 100-mL (\pm 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add 100 μ L of the solution containing the nine internal standards (Table 2).
- 13.2.2.3.4 Using appropriate methods, concentrate to approximately 10 mL.
- 13.2.2.3.5 Just before analysis, add 10 μ L tetradecane recovery standard solution (Table 2), and reduce the volume to a final volume of 20 μ L, as necessary. No column chromatography is required.
- 13.2.2.3.6 Analyze an aliquot following the same procedures used to analyze samples.
- 13.2.2.3.7 Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in pg-mL of rinsate solvent.

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13.3 Surrogate spiked into samples. A surrogate compound ^{37}Cl -2,3,7,8-TCDD may be spiked into all samples and QC samples for this method. The spike concentration is 1.0 ng per sample. This surrogate is spiked following extraction and just prior to cleanup as a "cleanup recovery standard", in order to monitor relative loss of internal standard during both extraction and cleanup.

13.4 Duplicates

13.4.1 In each batch of samples, locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration.

13.4.1.1 The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results.

13.4.1.2 Recommended actions to help locate problems:

13.4.1.2.1 Verify satisfactory instrument performance.

13.4.1.2.2 If possible, verify that no error was made while weighing the sample portions.

13.4.1.2.3 Review the analytical procedures with the performing laboratory personnel.

13.5 Matrix Spike and Matrix Spike Duplicate (MS and MSD)

13.5.1 Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

13.5.2 Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

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13.5.3 Analyze the MS and MSD samples as described in Section 12.

13.5.4 The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) must agree within 20 percent relative difference.

13.6 Percent Recovery of the Internal Standards

For each sample, method blank and rinsate, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards.

NOTE: A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data.

13.7 Identification Criteria

13.7.1 If either one of the identification criteria appearing in Sections 12.4.1.1 through 12.4.1.4, is not met for an homologous series, it is reported that the sample does not contain unlabeled 2,3,7,8-substituted PCDD/PCDF isomers for that homologous series at the calculated detection limit.

13.7.2 If the first initial identification criteria are met, but the criteria appearing in Sections 12.4.1.5 and 12.4.2.1, are not met, that sample is presumed to contain interfering contaminants. This must be noted on the analytical report form, and the sample should be rerun or the extract reanalyzed.

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14. CALCULATIONS

- 14.1 For gas chromatographic peaks that have met the criteria outlined in Section 12.4, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RRF}(n)}$$

where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

A_x = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = Sample size in g (if solid) or L (if liquid).

$\overline{RRF}(n)$ = Calculated mean relative response factor for the analyte [$\overline{RRF}(n)$ with $n = 1$ to 17; Section 9.1.4.7, this exhibit].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, $\overline{RRF}(n)$ is the value calculated using the equation in Section 9.1.4.7. However, if it is a non-2,3,7,8-substituted congener, the $\overline{RRF}(k)$ value is the one calculated using the equation in Section 9.1.4.8.2 [$\overline{RRF}(k)$ with $k = 27$ to 30.].

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- 14.2 Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Percent Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RRF}(m)} \times 100$$

where

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

Q_{is} = Quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = Quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RRF}(m)$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Section 9.1.4.9 [$\overline{RRF}(m)$ with $m = 18$ to 26].

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14.3 If the concentration in the 10-uL or 50-uL final extraction of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limits (MCL) listed in Table 1 (e.g., 200 pg/uL for TCDD in a 10g soil), the linear range of response versus concentration may have been exceeded, a reanalysis of the sample (using one tenth aliquot) should be undertaken. The volumes of the internal and recovery standard solutions should remain the same as described for the sample preparation (this exhibit, Section 11.1 to 11.9.2). For the other congeners (including OCDD), however, report the measured concentration and indicate that the value exceeds the MCL.

14.4 The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

14.5 Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

14.5.1 Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

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14.5.1.1 Use the expression for EDL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N < 2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ¹³C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their internal standard must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$\text{EDL (specific 2,3,7,8-subst. PCDD/PCDF)} = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times \overline{\text{RRF}}(n)}$$

where

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

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H_{is} = height of one of the quantitation ions (Table 6) for the labeled internal standards.

W , $\overline{RRF}(n)$, and Q_{is} retain the same meanings as defined in Section 14.1.

14.5.2 Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

14.5.2.1 When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.4, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 14.1, except that A_X in Section 14.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDL can be calculated using the above formula and the height of one of the ions as appropriate.

14.6 The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2) / 2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

14.7 The 2,3,7,8-TCDD toxic equivalents (TE) of PCDDs and PCDFs present in the sample are calculated, only at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the fifteen 2,3,7,8-substituted PCDDs and PCDFs (Table 3 and the non-2,3,7,8-substituted compounds as shown in Table 11). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 11. The exclusion of other homologous series such as mono-, di-, and tri- and chlorinated dibenzodioxins and dibenzofurans does not mean that they are non-toxic.

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Their toxicity, as known at this time, is much less than the toxicity of the compounds listed in Table 11. The above procedure for calculating the 2,3,7,8-TCDD toxic equivalents is not claimed by the CDWG to be based on a thoroughly established scientific foundation. The procedure, rather, represents a "Consensus recommendation on science policy". Since the procedure may be changed in the future, reporting requirements for PCDD and PCDF data would still include the reporting of the analyte concentrations of the PCDD/PCDF congener as calculated in Sections 14.1 and 14.4.

14.7.1 Two-GC Column TEF Determination

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60-m DB-5 GC column. In order to determine the proper concentrations of the individual 2,3,7,8-substituted congeners, the sample extract may be reanalyzed on another GC column.

14.7.1.1 The concentration of 2378-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq 25\%$ valley.

14.7.1.2 For samples that have a positive result for 2378-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2378-TCDF is then the result calculated from the DB-225 analysis. The chromatographic separation between 2378-TCDF and any other unlabeled TCDF isomers must be $\leq 25\%$ valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in section 14.1 through 14.6 (this exhibit).

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14.7.1.3 For samples that have positive results for the 2378-substituted penta and hexa isomers, the extract can be analyzed on either the 30m DB-225 column or a 60m SP-2330 (or SP-2331) column, if requested by the client. The GC/MS conditions are altered so that only the second and third descriptor (Table 6) are used. Concentration calculations are performed as in section 14.1 through 14.6 (this exhibit).

NOTE: The confirmation and quantitation of 2,3,7,8-TCDD (this exhibit, Section 14.7.1.1) may be accomplished on the SP-2330 GC column instead of the DB-5 column, provided the criteria listed in Section 8.1.2 (this exhibit) are met and the requirements described in Section 13.1 (this exhibit) are followed.

14.7.1.4 For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 12.4.2, this exhibit) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 12.4.1.1 (this exhibit) applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 12 and the results from the routine calibration run on the DB-5 column.

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**STANDARD
OPERATING
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This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipers and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

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EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 uL (either in a minivial or in a capillary tube). Inject two uL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 12 (this exhibit). Perform calculations according to Section 14 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is $25 \times 5 = 125$ pg/WTE and the positive response for the blank would be $8 \times 5 = 40$ pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

At a minimum, wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbant (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

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TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based
 Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Fish Tissue	Human Adipose Tissue	Sludges, Fuel Oil	Still- Bottom
Lower MCL ^a	0.02	2.0	2.0	2.0	2.0	10	20
Upper MCL ^a	4.0	400	400	400	400	2000	4000
Weight (g)	1000	10	10	10	10	2.0	1.0
IS Spiking Levels (ng)	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Final Extr. Vol.(uL)	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF/PeCDD/PeCDF, by 2.5 for HxCDD/HxCDF/HpCDD/HpCDF, and by 5 for OCDD/OCDF.

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TABLE 2

COMPOSITION OF THE SAMPLE FORTIFICATION
AND RECOVERY STANDARD SOLUTIONS

Analyte	Sample Fortification Solution Concentration (pg/uL; Solvent: Isooctane)	Recovery Standard Solution Concentration (pg/uL; Solvent: Tetradecane)
¹³ C-2,3,7,8-TCDD	10	--
¹³ C-2,3,7,8-TCDF	10	--
¹³ C-1,2,3,4-TCDD	--	100
¹³ C-1,2,3,7,8-PeCDD	10	--
¹³ C-1,2,3,7,8-PeCDF	10	--
¹³ C-1,2,3,6,7,8-HxCDD	25	--
¹³ C-1,2,3,4,7,8-HxCDF	25	--
¹³ C-1,2,3,7,8,9-HxCDD	--	100
¹³ C-1,2,3,4,6,7,8-HpCDD	25	--
¹³ C-1,2,3,4,6,7,8-HpCDF	25	--
¹³ C-OCDD	50	--

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TABLE 3
THE FIFTEEN 2,3,7,8-SUBSTITUTED PCDD AND PCDF CONGENERS

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF

(*)The ¹³C-labeled analog is used as an internal standard.
 (+)The ¹³C-labeled analog is used as a recovery standard.

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TABLE 4

ISOMERS OF CHLORINATED DIOXINS AND FURANS AS A
 FUNCTION OF THE NUMBER OF CHLORINE ATOMS

Number of Chlorine Atoms	Number of Dioxin Isomers	Number of 2,3,7,8 Isomers	Number of Furan Isomers	Number of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	75	7	135	10

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TABLE 5

HIGH-RESOLUTION CONCENTRATION CALIBRATION SOLUTIONS

		<u>Concentration (pg/uL, in Nonane)</u>				
Compound	HRGCC	5	4	3	2	1
Unlabeled Analytes						
2,3,7,8-TCDD		200	50	10	2.5	1
2,3,7,8-TCDF		200	50	10	2.5	1
1,2,3,7,8-PeCDD		500	125	25	6.25	2.5
1,2,3,7,8-PeCDF		500	125	25	6.25	2.5
2,3,4,7,8-PeCDF		500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDD		500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDD		500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDD		500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDF		500	125	25	6.25	2.5
2,3,4,6,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDD		500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDF		500	125	25	6.25	2.5
1,2,3,4,7,8,9-HpCDF		500	125	25	6.25	2.5
OCDD		1000	250	50	12.5	5
OCDF		1000	250	50	12.5	5
Internal Standards						
¹³ C ₁₂ -2,3,7,8-TCDD		50	50	50	50	50
¹³ C ₁₂ -2,3,7,8-TCDF		50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDD		50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDF		50	50	50	50	50
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD		125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF		125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD		125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF		125	125	125	125	125
¹³ C ₁₂ -OCDD		250	250	250	250	250
Recovery Standards						
¹³ C ₁₂ -1,2,3,4-TCDD(a)		100	100	100	100	100
¹³ C ₁₂ -1,2,3,4,7,8,9-HxCDD(b)		100	100	100	100	100

(a) Used for recovery determination of TCDD, TCDF, PeCDD and PeCDF internal

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TABLE 6

IONS MONITORED FOR HRGC/HRMS ANALYSIS OF PCDD/PCDFs
 ((S) = internal/recovery standard)

Descriptor	Accurate(a) Mass	Ion ID	Elemental Composition	Analyte
1	303.9016	M	CH ³⁵ Cl	TCDF
	305.8987	M+2	CH ³⁵ Cl ³⁷ ClO	TCDF
	315.9419	M	CH ³⁵ ClO	TCDF (S)
	317.9389	M+2	¹³ CH ³⁵ Cl ³⁷ ClO	TCDF (S)
	319.8965	M	CH ³⁵ ClO	TCDD
	321.8936	M+2	CH ³⁵ Cl ³⁷ ClO	TCDD
	331.9368	M	¹³ CH ³⁵ ClO	TCDD (S)
	333.9339	M+2	¹³ CH ³⁵ Cl ³⁷ ClO	TCDD (S)
	339.8597	M+2	CH ³⁵ Cl ³⁷ ClO	PeCDF
	341.8567	M+4	CH ³⁵ Cl ³⁷ ClO	PeCDF
	375.8364	M+2	CH ³⁵ ClO	HxCDFE
	316.9824	LOCK	CF	PFK
2	351.9000	M+2	¹³ CH ³⁵ Cl ³⁷ ClO	PeCDF (S)
	353.8970	M+4	¹³ CH ³⁵ Cl ³⁷ ClO	PeCDF (S)
	355.8546	M+2	CH ³⁵ Cl ³⁷ ClO	PeCDD
	357.8516	M+4	CH ³⁵ Cl ³⁷ ClO	PeCDD
	367.8949	M+2	¹³ CH ³⁵ Cl ³⁷ ClO	PeCDD (S)
	369.8919	M+4	¹³ CH ³⁵ Cl ³⁷ ClO	PeCDD (S)

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TABLE 6
 Continued

Descriptor	Accurate(a) Mass	Ion ID	Elemental Composition	Analyte
	339.8597	M+2	CH ³⁵ C ¹³⁷ C ¹⁰	PeCDF
	341.8567	M+4	CH ³⁵ C ¹³⁷ C ¹⁰	PeCDF
	409.7974	M+2	CH ³⁵ C ¹⁰	HpCDPE
	366.9793	LOCK	CF	PFK
3	373.8208	M+2	CH ³⁵ C ¹³⁷ C ¹⁰	HxCDF
	375.8178	M+4	CH ³⁵ C ¹³⁷ C ¹⁰	HxCDF
	383.8642	M	¹³ CH ³⁵ C ¹⁰	HxCDF (S)
	385.8610	M+2	¹³ CH ³⁵ C ¹³⁷ C ¹⁰	HxCDF (S)
	389.8156	M+2	CH ³⁵ C ¹³⁷ C ¹⁰	HxCDD
	391.8127	M+4	CH ³⁵ C ¹³⁷ C ¹⁰	HxCDD
	401.8559	M+2	¹³ CH ³⁵ C ¹³⁷ C ¹⁰	HxCDD (S)
	403.8529	M+4	¹³ CH ³⁵ C ¹³⁷ C ¹⁰	HxCDD (S)
	445.7555	M+4	CH ³⁵ C ¹³⁷ C ¹⁰	OCDPE
	380.9760	LOCK	CF	PFK
4	407.7818	M+2	CH ³⁵ C ¹³⁷ C ¹⁰	HpCDF
	409.7789	M+4	CH ³⁵ C ¹³⁷ C ¹⁰	HpCDF
	417.8253	M	¹³ CH ³⁵ C ¹⁰	HpCDF (S)

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TABLE 6

Continued

Descriptor	Accurate(a) Mass	Ion ID	Elemental Composition	Analyte
	419.8220	M+2	$^{13}\text{CH}^{35}\text{C}^{137}\text{ClO}$	HpCDF (S)
	423.7766	M+2	$\text{CH}^{35}\text{C}^{137}\text{ClO}$	HpCDD
	425.7737	M+4	$\text{CH}^{35}\text{C}^{137}\text{ClO}$	HpCDD
	435.8169	M+2	$^{13}\text{CH}^{35}\text{C}^{137}\text{ClO}$	HpCDD (S)
	437.8140	M+4	$^{13}\text{CH}^{35}\text{C}^{137}\text{ClO}$	HpCDD (S)
	479.7165	M+4	$\text{CH}^{35}\text{C}^{137}\text{ClO}$	NCDPE
	442.9730	LOCK	CF	PFK
5	441.7428	M+2	$\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDF
	443.7399	M+4	$\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDF
	457.7377	M+2	$\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDD
	459.7348	M+4	$\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDD
	469.7779	M+2	$^{13}\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDD (S)
	471.7750	M+4	$^{13}\text{C}^{35}\text{C}^{137}\text{ClO}$	OCDD (S)
	513.6775	M+4	$\text{C}^{35}\text{C}^{137}\text{ClO}$	DCDPE
	454.9728	LOCK	CF	PFK

(a) The following nuclidic masses were used:

H = 1.007825	O = 15.994915
C = 12.000000	^{35}Cl = 34.968853
^{13}C = 13.003355	^{37}Cl = 36.965903

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TABLE 7

RECOMMENDED GC OPERATING CONDITIONS

The GC Operating Conditions (Temperatures (°C), and Times (minutes))
Are as Follows:

Injector Temperature: 280°C

Interface Temperature: 280°C

Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

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TABLE 8
PCDD AND PCDF CONGENERS PRESENT IN THE GC PERFORMANCE
EVALUATION SOLUTION AND USED FOR DEFINING THE HOMOLOGOUS
GC RETENTION TIME WINDOWS ON A 60-M DB-5 COLUMN

No. of Chlorine Atoms	PCDD-Positional Isomer		PCDF-Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4(a)	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/ 1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

(a) In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-,
 1,2,3,8-, 2,3,7,8-, ¹³C₁₂-2,3,7,8-, and 1,2,3,9-TCDD isomers
 must also be present.

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TABLE 9
 THEORETICAL ION ABUNDANCE RATIOS AND THEIR CONTROL LIMITS
 FOR PCDDs AND PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			lower	upper
4	$\frac{M}{M+2}$	0.77	0.65	0.89
5	$\frac{M+2}{M+4}$	1.55	1.32	1.78
6	$\frac{M+2}{M+4}$	1.24	1.05	1.43
6 ^(a)	$\frac{M}{M+2}$	0.51	0.43	0.59
7 ^(b)	$\frac{M}{M+2}$	0.44	0.37	0.51
7	$\frac{M+2}{M+4}$	1.04	0.88	1.20
8	$\frac{M+2}{M+4}$	0.89	0.76	1.02

^(a) Used only for ¹³C-HxCDF (IS).

^(b) Used only for ¹³C-HpCDF (IS).

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TABLE 10
 RELATIVE RESPONSE FACTOR [RRF (number)] ATTRIBUTES

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCDF
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	¹³ C ₁₂ -2,3,7,8-TCDD
19	¹³ C ₁₂ -2,3,7,8-TCDF
20	¹³ C ₁₂ -1,2,3,7,8-PeCDD
21	¹³ C ₁₂ -1,2,3,7,8-PeCDF
22	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD
23	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF
24	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD
25	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF
26	¹³ C ₁₂ -OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

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TABLE 11
2,3,7,8-TCDD EQUIVALENT FACTORS (TEFs) FOR THE
POLYCHLORINATED DIBENZODIOXINS AND DIBENZOFURANS

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	1,2,3,4,6,7,8,9-OCDD	0.001
8	2,3,7,8-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8-PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	1,2,3,4,6,7,8,9-OCDF	0.001

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TABLE 12
TOXICITY EQUIVALENCY FACTOR: ANALYTE RELATIVE
RETENTION TIME REFERENCE ATTRIBUTES

Analyte	Analyte RRT Reference(a)
1,2,3,4,7,8-HxCDD	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}\text{C}_{12}$ -1,3,7,8-PeCDF, and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF.

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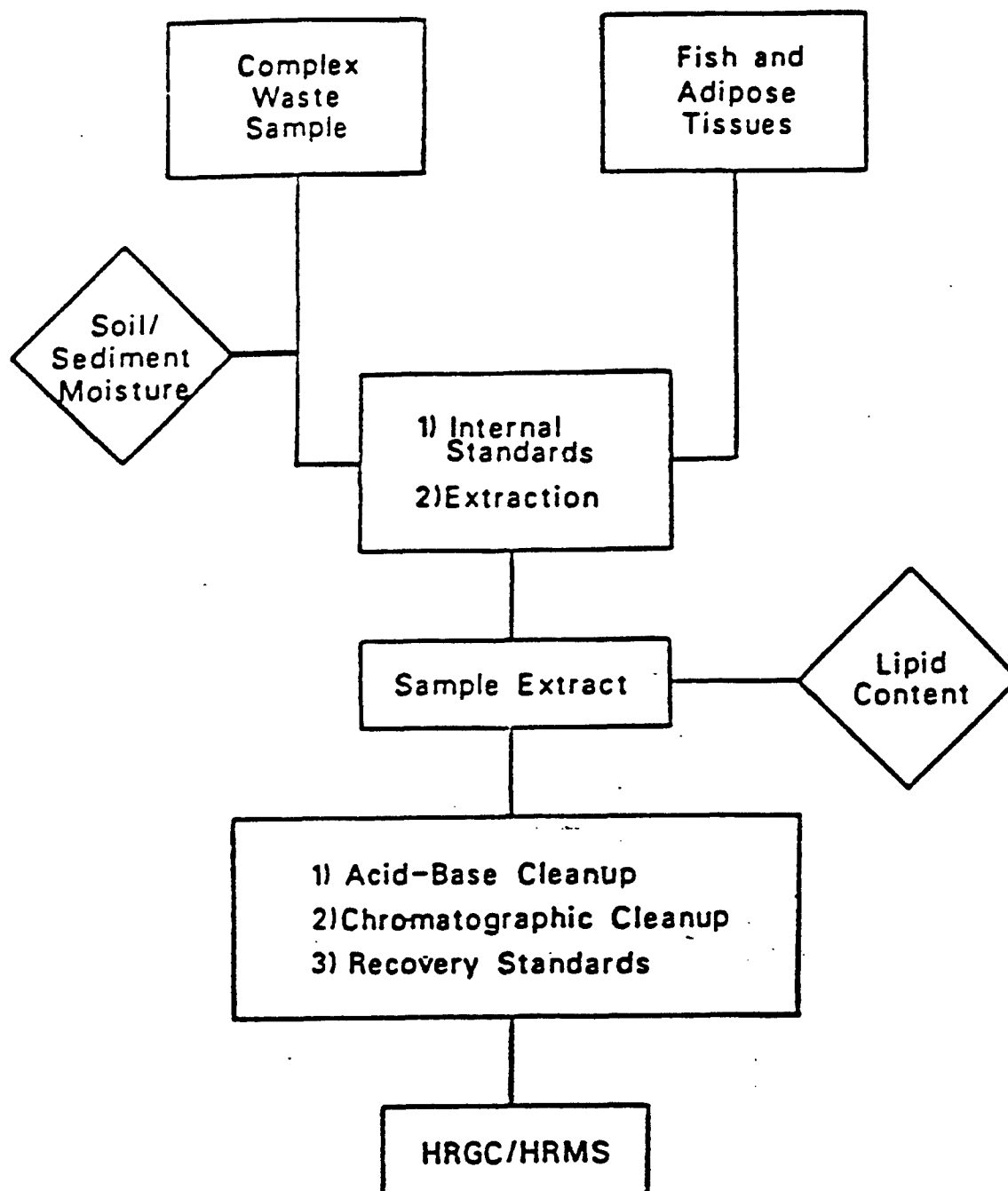


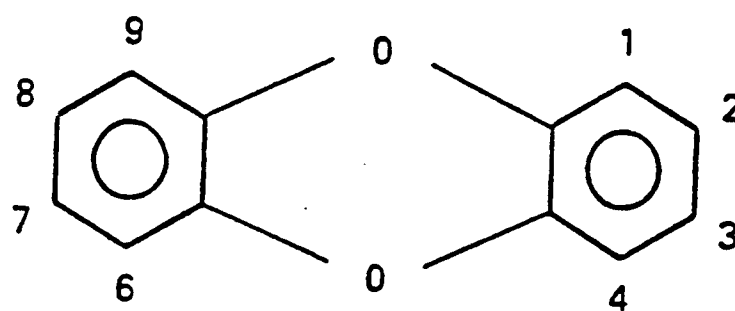
Figure 1

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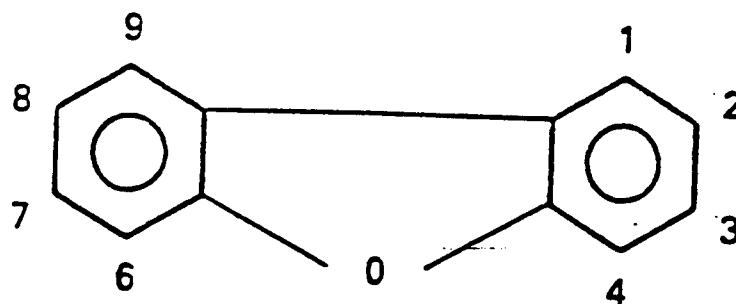
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Dibenzodioxin



Dibenzofuran

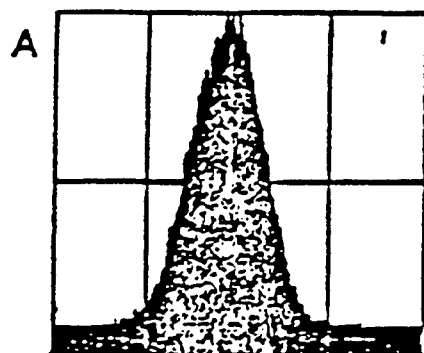
Figure 2

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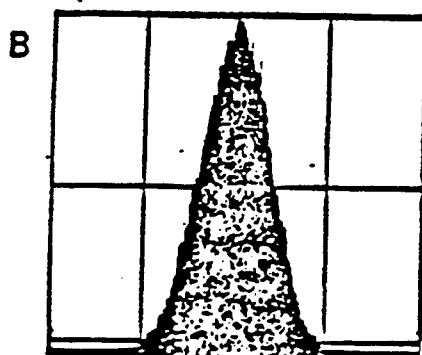
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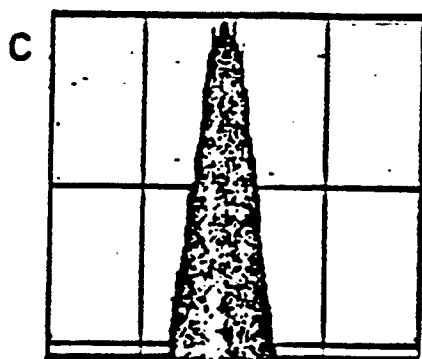
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5,600



5,600



8,550

← 400 ppm →

Figure 3

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Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS

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Analytical Procedure

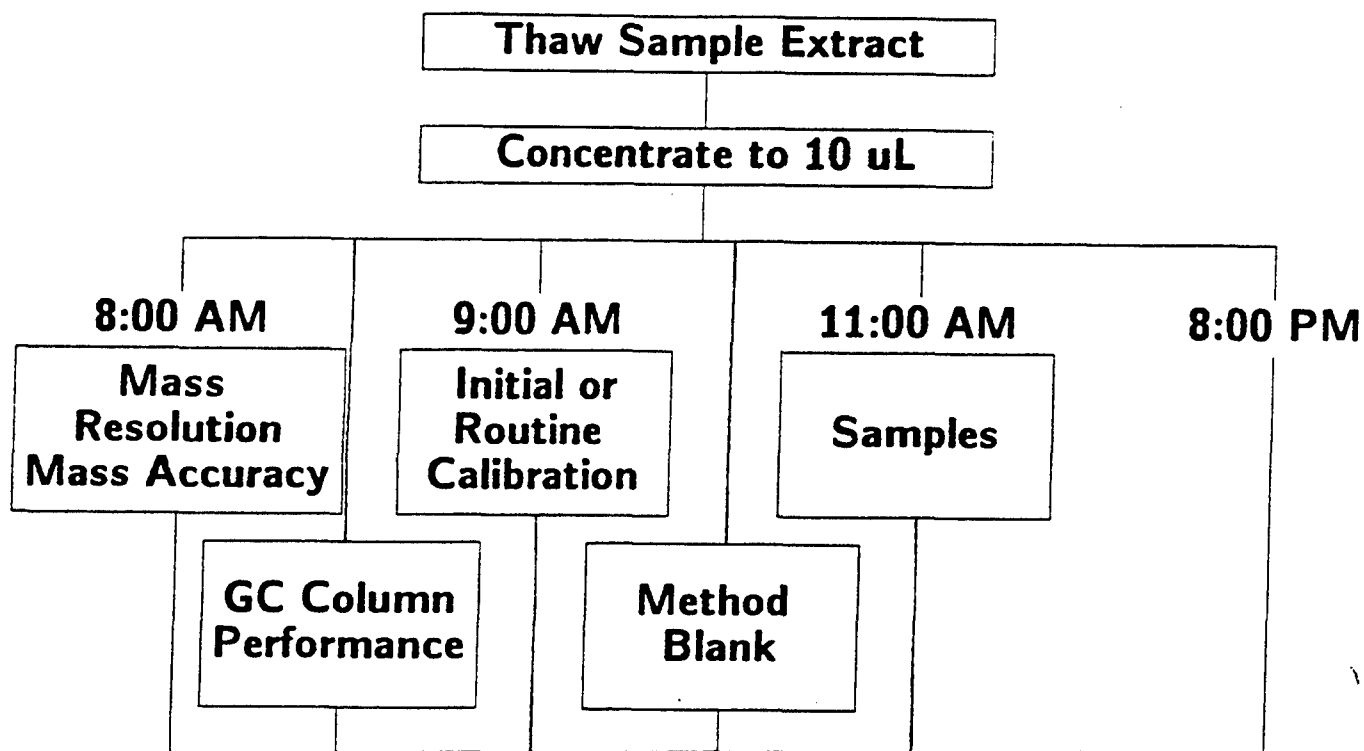


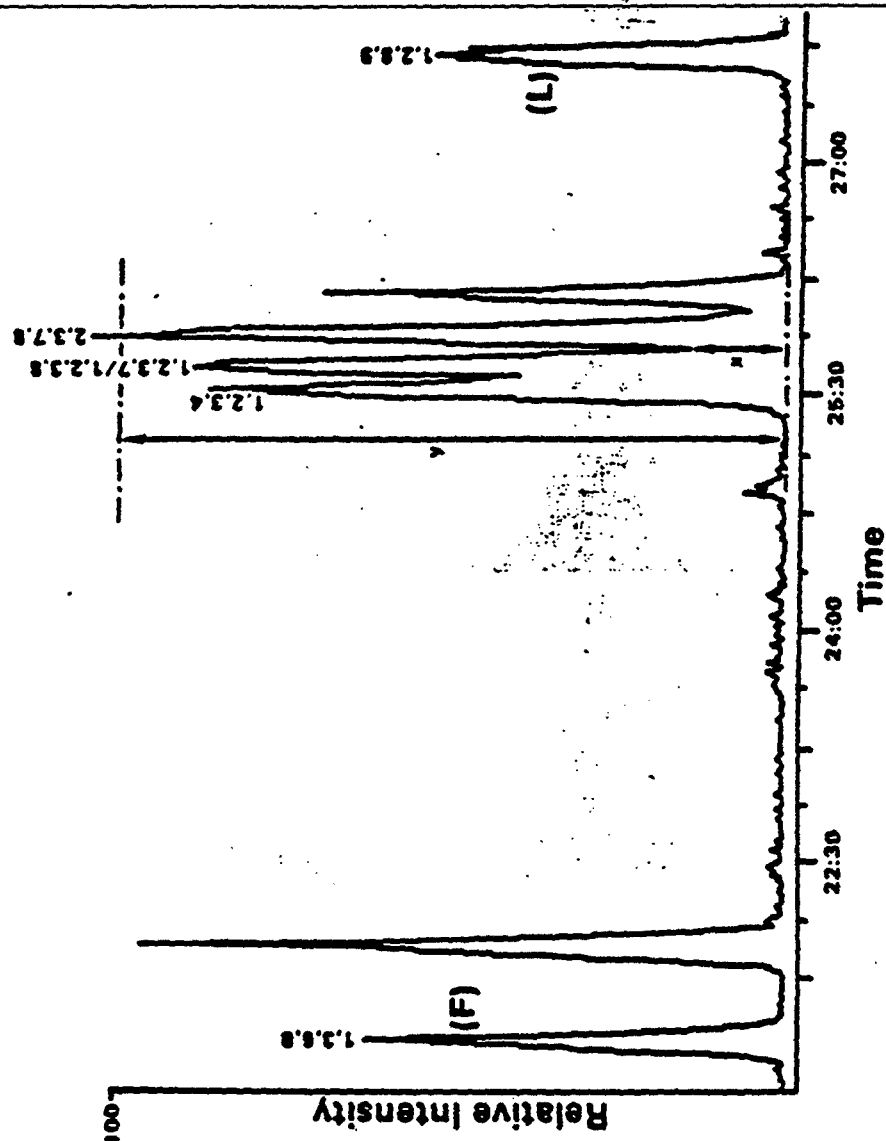
Figure 4

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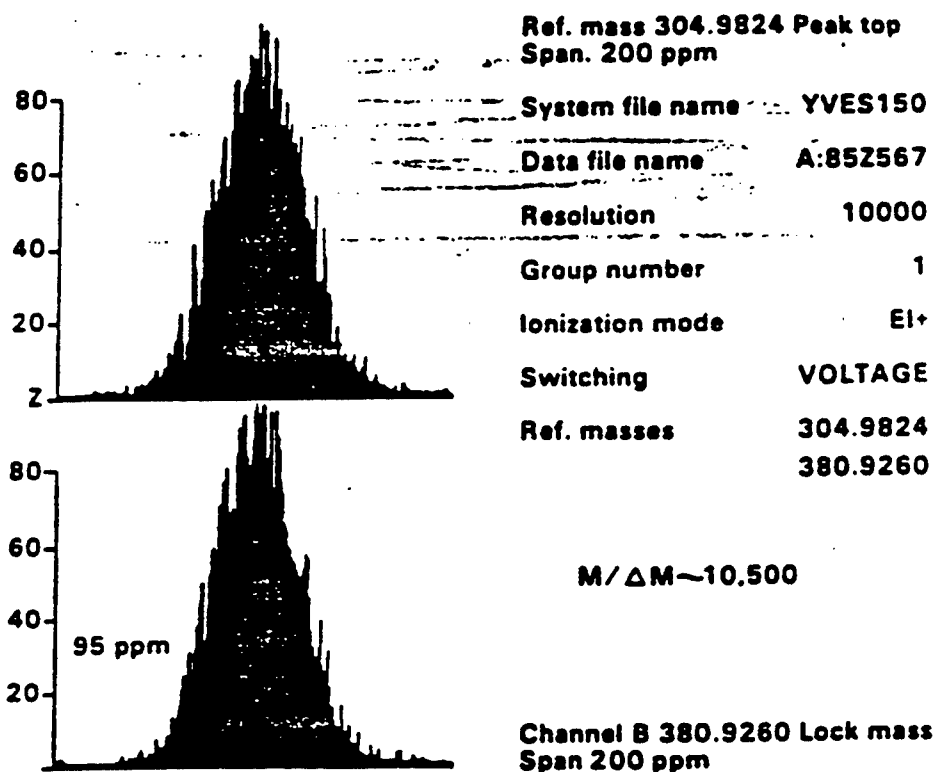
Figure 5

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Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS

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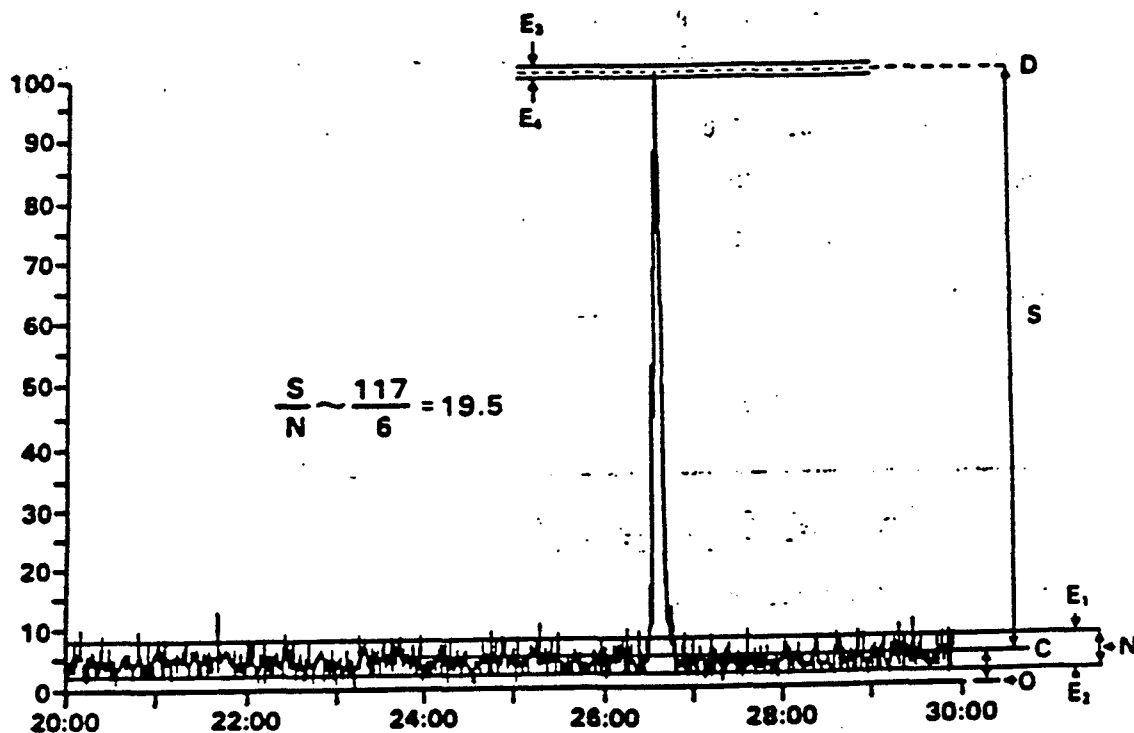
Peak profiles representing two PFK reference ions at m/z 305 and 381. The resolution of the high-mass signal is 95 ppm at 5 percent of the peak height; this corresponds to a resolving power M/ΔM of 10,500 (10 percent valley definition).

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Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

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Figure 7

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

**TITLE: THE DETERMINATION OF TOTAL PETROLEUM (FUEL)
HYDROCARBONS IN WATER AND SOIL SAMPLES BY GAS
CHROMATOGRAPHY (MODIFIED EPA 8015)**

Effective Date: 3-27-96

Prepared by: Bradley A. Weichert

BA Weichert 3/27/96

Reviewed by: Michael G. Winslow

Michael G. Winslow 3/27/96

Approved by: John J. Mousa
(Gainesville Laboratory Director)

JJM 3/27/96

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- 2.0 SCOPE AND APPLICATION**
- 3.0 SUMMARY OF METHOD**
- 4.0 APPARATUS AND MATERIALS**
- 5.0 METHOD INTERFERENCES**
- 6.0 SAFETY PRACTICES**

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**TITLE: THE DETERMINATION OF TOTAL PETROLEUM (FUEL)
HYDROCARBONS IN WATER AND SOIL SAMPLES BY GAS
CHROMATOGRAPHY (MODIFIED EPA 8015)**

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the method used to determine total petroleum (fuel) hydrocarbons in water and soil samples by gas chromatography using a flame ionization detector.

2.0 SCOPE AND APPLICATION

This method is applicable to jetfuels, aviation gasoline, automotive gasoline, and diesel fuels, in both water and soil samples.

3.0 SUMMARY OF METHOD

Petroleum hydrocarbons are extracted from water and soil samples using carbon disulfide (CS₂). The extract is then analyzed by capillary gas chromatography with a flame ionization detector (GC/FID). The elution pattern and area response of the chromatogram is compared to that of standards (various fuels). It is possible to determine the type of fuel present, i.e., gasoline, diesel, etc., provided the source material has not been subjected to extensive weathering.

4.0 APPARATUS AND MATERIALS

- 4.1 Gas Chromatograph - A gas chromatograph (HP5890A or equivalent) equipped with a flame ionization detector, capillary injector and capable of temperature programming is used. A computerized data system (Nelson Analytical or equivalent) is used to collect the chromatographic data.

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- 4.2 The gas chromatographic column used is a 30m x 0.25mm fused silica column coated with DB-17 or DB-5 (0.25um film). This column is manufactured by J&W Scientific.

Operating Conditions:

Injector Temperature: 250°C
Detector Temperature: 300°C
Column Temperature Program: 40°C/10 min to 280°C @ 10°C per min.,
hold 4 min

- 4.3 Two microliter samples are injected using an HP7672 autosampler operated using a Grob type injection.
- 4.4 The data is collected and stored into a Nelson 2700 data system. The data system is capable of reprocessing chromatographic data, comparing chromatograms and measuring peak areas. Data can be stored for future reference using most electronic storage media.
- 4.5 Surrogate spike solution - A surrogate spike solution of n-pentacosane at 2400 ug/mL in acetone. Spike 200 uL to each soil sample, and 100 uL to each water sample.

5.0 METHOD INTERFERENCES

- 5.1 Interferences in the trace level determination of substances can originate from numerous sources. The FID detector is not selective and will detect many volatile organic compounds. Contamination can arise from the matrix in which the sample is found, the cleanliness of glassware and the care in which the sample is handled by laboratory personnel.
- 5.2 Samples can also be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent handling can serve as a check on such contamination.
- 5.3 This method will detect most hydrocarbons that are partitioned and extracted into carbon disulfide.

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- 5.4 "Fresh" fuels exhibit characteristic chromatographic patterns that can be readily identified if there is no significant weathering due to evaporation, dissolution or microbial degradation. Interferences due to non-petroleum related hydrocarbons (eg. PCBs) can result in a positive interference that can be difficult to discern due to petroleum's complex chromatographic patterns. Only analysts experienced in the analysis of petroleum hydrocarbons should perform this method.

6.0 SAFETY PRACTICES

Carbon disulfide is highly toxic and extremely FLAMMABLE. All open containers, vials, etc., must be handled in a fume hood. The hood must not contain any heat sources, e.g., functioning hot plates.

7.0 HOLDING TIMES

Soil or preserved water samples should be extracted and analyzed within 14 days of sample extraction. Water samples are preserved by adjustment of pH to <2 , otherwise holding time is only 7 days. Water samples are collected and held in 120 mL amber glass small mouth bottles with teflon lined septa and with zero head-space.

8.0 STANDARDS PREPARATION

- 8.1 Standards should be prepared from serial, volumetric dilutions from the type of fuel expected in the sample for quantitation. Ideally, source material, if available, should be used due to the formulation differences of fuels from seasonal requirements and raw source materials.
- 8.2 Stock standards are prepared gravimetrically in CS_2 . All subsequent dilutions are prepared with CS_2 . Standards should be prepared to encompass the range of response expected in the samples. The lowest standard of fuel should be 8 ug/mL or less. A three order of magnitude curve should be sufficient for the analysis of most environmental samples. The surrogate is added to all standards.

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COMPOUND	LOWER STANDARD (ug/mL)	UPPER STANDARD (ug/mL)	CERTIFIED REPORTING LIMIT	
			AQUEOUS	SOLID
GAS	8	4000	0.4 mg/L	8 ug/g
DIESEL	8	4000	0.4 mg/L	8 ug/g
PENTACOSANE (SURROGATE)	1	125	NA	

COMPOUND	NOMINAL CALIBRATION STANDARD CONCENTRATIONS (ug/mL)					
	STD A	STD B	STD C	STD D	STD E	STD F
GAS	4000	800	400	200	40	8
DIESEL	4000	800	400	200	40	8
PENTACOSANE (SURROGATE)	125	100	50	25	5	1

8.3 External standard calibration is used to prepare a curve to compare the response of the standards with the response of the samples. The extract concentration is calculated from the curve and the final concentration is corrected for extract volume, sample volume, and dilutions. Results for soil samples are also corrected for % moisture content.

8.4 Chromatographic patterns are matched to the fuel type by a variety of factors. The characteristic identifier of a petroleum fuel is its boiling envelope. The gasoline range is from ca. C₅ to C₁₀. The diesel range is from ca. C₁₀ to C₂₂. The jet fuels JP-4 and JP-5 exhibit a more refined envelope in the diesel envelope.

9.0 PROCEDURE

9.1 A measured quantity or aliquot of sample is transferred to the extraction vessel. Surrogate is added to each sample prior to mixing.

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- 9.1.1 For soil samples: A 10 g soil sample is transferred to a 40 or 60 mL narrow mouth screw top bottle. Ten milliliters of CS₂ is added to the bottle and sealed with a teflon lined septum screw cap. The sample is placed on a reciprocating shaker and vigorously shaken for 30 minutes. A 1 mL aliquot is then placed in a crimp sealed 1 mL autosampler vial. The sample is then analyzed by GC/FID.
- 9.1.2 For water samples: Shake the 120 mL bottle of sample. Remove a 10 mL aliquot, with a syringe, from the middle of the sample and discard. Immediately add 5 mL of CS₂ and mechanically tumble for 1 hour to extract. Remove the CS₂ and place in a sealed amber vial for analysis. After the extraction is complete, measure the volume of sample in the bottle and subtract 10 mL to obtain the sample volume.
- 9.2 The gas chromatograph is set-up according to the analytical conditions specified in Section 4.0 and the samples are loaded in the autosampler for analysis.
- 9.3 The external calibration technique is used to calibrate the GC system.
- 9.4 Retention time windows must be established for all analytes and for all columns used for analysis.
- 9.5 Continuing Calibration Check Standard (CCS) -- The analyst must analyze the continuing calibration check standard at minimum intervals of every 10 samples and at the end of the run. The response of the CCS should be within 15 percent of the same standard in the calibration curve. If the continuing calibration fails the requirements, the analyst must follow the corrective actions outlined in the QA/QC Manual before continuing with the analysis. The analyst must check the response of the target analytes within each sample. If the response of an analyte exceeds the calibration range for that analyte the sample must be diluted and reanalyzed.

10.0 CALCULATIONS

10.1 Summary

The target responses are transferred to the Laboratory Data Management System, CLASS (Chemical Laboratory Analytical and Scheduling System),

along with any relevant sample information. The concentration is calculated using the regression equation calculated by CLASS. Final sample results are corrected for sample volume or sample weight, extract volume, percent moisture for solid samples, dilution factors and any applicable conversion factors.

10.2 Peak Identification

Analysis identification of fuels depends primarily on the pattern matching and boiling range (envelope). For analyses requesting both diesel and gas, a combined standard curve is used. Prior to initial calibration, the analyst determines the standard pattern for the individual fuels and determines where peak overlap occurs. The analyst will integrate total area for all non-overlapping peaks. Analyte response that lies within the established retention time windows will be considered to be tentatively identified. Analyst must use their judgement as to whether the peaks may represent a target compound by examining such factors as a peak pattern and resolution from interferences and matrix "noise". For analyses of samples for one fuel only, and individual curve can be run. Total area under the peaks will be used for quantitation if the peak pattern is determined to match that of the target fuel.

10.3 Analyte Quantitation

The sum of total area under the peaks is used to calculate analyte concentrations.

11.0 QUALITY CONTROL

11.0 In order to demonstrate method performance, the analysis of spiked samples will be performed during the extraction and analysis of environmental samples. At a minimum, standard ESE quality assurance procedures should be followed. For each lot of samples, extract and analyze the following QC required unless specified differently by the client:

5%	Method Blank
5%	Standard Matrix Spike
5%	Sample Matrix Spike
5%	Sample Matrix Spike Duplicate
100%	Samples Spiked With Surrogate

Note: The number of QC required is the actual number of QC samples rounded up to

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the nearest whole number, i.e, 5% = 1 QC for 1-20 samples; 2 QC for 21-40
For each batch of 20 samples, one standard matrix spike, one sample matrix spike, and one sample matrix spike duplicate analysis should be performed.

- 11.1 If the type of fuel to be expected in the samples is known, then that type of fuel should be used in the preparation of spike solutions. If the type of fuel is not known, then a diesel stock spike solution should be prepared in acetone. If i.e. gas and/or diesel are being evaluated, prepare separate solutions for spikes of each.
- 11.2 Spike solution should be prepared in acetone at a concentration (40000 ug/mL) sufficient to give a significant response above the background for the matrix spike analyses. One hundred microliters of spike solution should be used to spike soil samples, and 50 microliters for water samples.

COMPOUND	SPIKE SOLUTION CONC. (ug/mL)	SPIKE VOLUME (mL)		FINAL EXTRACT VOLUME (mL)		TARGET CONCENTRATION IN EXTRACT	
		AQUEOUS	SOLID	AQUEOUS	SOLID	AQUEOUS	SOLID
GAS	40000	0.05	0.1	5	10	400	400
DIESEL	40000	0.05	0.1	5	10	400	400
PENTACOSANE (SURROGATE)	2400	0.1	0.2	5	10	48	48

- 11.3 The corrective action procedures that will be taken following a failure to meet QC criteria are listed in Table 13-5 of the LCQAP.

12.0 REFERENCE

- 12.1 EPA Method 8015 -- Test Methods for Evaluating Solid Wastes, EPA SW 846 3rd Edition, September 1986.

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Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Liquid Pressure Chromatography

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.
Continuing calibration standard (CCS)	+/- 15% of standard initial response for GC (except for NPD which is +/-25%) and +/- 10% of standard initial response for HPLC	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples when last CCS is acceptable, or document why data are acceptable.
Method blank (MB)	< than two times DL for nonvolatile organics (listed in reporting limit tables in Section 5)	Determine and correct cause of the blank problem, reanalyze the samples, if necessary, or document why data are acceptable.
Method blank (MB)	No greater than five times DL (listed in reporting limit tables in Section 5 for methylene chloride, acetone, toluene, and xylene organics. All other analytes must be \leq two times DL (listed in reporting limit tables in Section 5.	Reanalyze another MB. If second MB exceeds criteria, clean and recalibrate analytical system or document why data are acceptable.

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Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography (Continued, Page 2 of 3)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See precision and accuracy tables in section 5 for percent recovery control limits	Determine and correct the problem, reanalyze samples if necessary, or document why data are acceptable.
Sample matrix spike	See precision and accuracy tables in section 5 for percent recovery control limits	If standard matrix spike analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
Sample matrix spike duplicate	See precision and accuracy tables in section 5 for RPD control limits	If standard matrix analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
Surrogates* (SUR)	See tables in section 5 for percent recovery control limits	If surrogates in the MB or SP are within control limits, qualify data. If not, reanalyze samples with surrogates outside criteria or document why data are acceptable.

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Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography (Continued, Page 2 of 3)

Note: DL = detection limit.
GC = gas chromatography.
HPLC = high pressure liquid chromatography.
NPD = nitrogen-phosphorus detector.
RPD = relative percent difference.

*Surrogate/surrogates will only be spiked in samples if specified by the method.

Source: ESE

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

TITLE: **GAS CHROMATOGRAPHIC ANALYSIS OF CHLORINATED
HERBICIDES IN WATER AND SOIL (EPA METHODS 615 AND
8150)**

Effective Date: 9/11/95

Prepared by: Brad A. Weichert

Brad A. Weichert 8/8/95

Reviewed by: Mike G. Winslow

Mike G. Winslow 8/10/95

Approved by: John J. Mousa
(Gainesville Laboratory Director)

John J. Mousa 8/10/95

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10.0 SAFETY PRACTICES

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**TITLE: GAS CHROMATOGRAPHIC ANALYSIS OF CHLORINATED
HERBICIDES IN WATER AND SOIL (EPA METHODS 615 AND
8150)**

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a consistent method for the analysis of certain chlorinated herbicides in industrial and municipal wastewater and soils.

2.0 SCOPE

2.1 This SOP is a gas chromatographic (GC) method used to determine certain chlorinated herbicides. The following compounds can be determined by this method:

Compound Name	Cas No. ^a
2,4-D	94-75-7
2,4-DB	94-82-6
2,4,5-T	93-76-5
2,4,5-TP (Silvex)	93-72-1
Dalapon	75-99-0
Dicamba	1918-00-9
Dichlorprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2

^a Chemical Abstract Services Registry Number

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- 2.2 When this SOP is used to analyze unfamiliar samples, compound identifications should be determined by at least one other qualitative technique. This SOP describes analytical conditions for a second gas chromatographic column that can be used to confirm the analysis made by the primary column.

3.0 SUMMARY OF METHOD

- 3.1 This SOP provides a method for the analysis of chlorinated acid herbicides by gas chromatogram (GC). Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. Prior to use of this method, the appropriate sample extraction must be used. The GC is calibrated with five standards that correspond to the expected range of the concentrations found in the samples. A 1 ul extract is analyzed by GC with an electron capture detector (ECD). The results are reported as the acid equivalents.
- 3.2 The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations.

4.0 METHOD INTERFERENCES

- 4.1 Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols, including chlorophenols, may also interfere with this procedure.
- 4.2 Alkaline hydrolysis and subsequent extraction of the basic solution remove many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis.
- 4.3 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware and glass wool must be acid-rinsed, and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

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5.0 APPARATUS AND MATERIALS

5.1 Gas Chromatograph

5.1.1 Hewlett Packard 5890 Gas Chromatograph or equivalent equipped with dual Ni-63 ECD's, dual auto injectors, and capable of temperature programming is used. The GC must be able to accommodate two dissimilar columns. A computerized data system (PE Nelson Turbochrom) is used to collect chromatographic data.

5.1.2 Columns

5.1.2.1 Analytical: DB-17 30 meter x 0.25 mm fused silica capillary column with 0.25 um film

5.1.2.2 Confirmation: DB-5 30 meter x 0.25 mm fused silica capillary column with 0.25 um film

5.2 1 ml clear vials

5.3 Seals

5.4 9" Pasteur pipets

5.5 Crimper

5.6 Volumetric flasks - 25, 50, 100 mls

5.7 Volumetric pipets - various sizes ranging from 0.5 to 20.0 mls.

5.8 Amber bottles - 25 and 50 ml.

5.9 Micropipets - 50, 100 and 200 ul.

5.10 Analytical balance

5.11 Spatula

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6.0 REAGENTS

6.1 Solvents

6.1.1 Methanol - Pesticide grade.

6.1.2 Diethyl Ether - Pesticide grade.

6.1.3 Acetone - Pesticide grade.

6.1.4 Hexane - Pesticide grade.

6.2 Stock Standard solutions - available from commercial vendors.

6.2.1 Certified solutions - EM Science.

6.3 Stock Surrogate - 2,4-Dichlorophenyl acetic acid, 99.9%, available from Aldrich or equivalent

7.0 PROCEDURE

7.1 Calibration Standards

7.1.1 Stock Standard solutions - Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions. Certified stock solutions are supplied by EM Science as mixed intermediate solutions at nominal concentrations of 1 mg/mL.

7.1.2 Working Standards - A minimum of five calibration standards for each parameter of interest should be prepared through dilution of the secondary stock with hexane. They should be prepared at the following recommended nominal concentrations:

<u>MCPA/MCPP</u> <u>(ng/mL)</u>	<u>ALL OTHER</u> <u>(ng/mL)</u>	<u>DCPA (surrogate)</u> <u>(ng/mL)</u>
200	20	40
300	30	60
400	40	80
1000	100	200

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2000

200

400

Working standards must be prepared every six months, or sooner, if comparison with a check standard indicates a problem.

7.1.3 Surrogate Standards - The analyst should monitor the performance of the extraction, cleanup (when used), analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard spike sample, sample matrix spike and reagent water blank with 2,4-Dichlorophenyl acetic acid. Certified surrogate spike solutions are supplied by EM Science at a nominal concentration of 1 mg/mL.

7.1.3.1 Secondary Stock Surrogate solution - Pipet 1 ml of primary stock surrogate solution into a 100 ml volumetric flask and fill to volume with methanol. Final concentration is 10 ug/ml (10,000 ng/ml) 2,4-Dichlorophenyl acetic acid.

7.1.3.2 Working solution - Pipet 5.0 mls of the secondary stock surrogate solution into a 50 ml volumetric flask and fill to volume with methanol. Final concentration 1,000 ng/ml. A 1 ml aliquot must be spiked into all samples and QC samples.

7.1.4 Stock Spiking solution - Spike solutions are supplied by EM Science at a nominal concentration of 1 mg/mL each.

7.1.4.1 Secondary Spiking solution - Pipet 1 ml of primary stock spiking solution (section 7.1.4) into a 100 ml volumetric flask and fill to volume with methanol. Final concentration is 10 ug/ml (10,000 ng/ml).

7.1.4.2 Working Spiking solution - Pipet 2.5 mls of the Secondary Spiking solution (section 7.1.4.1) into a 100 ml volumetric flask and dilute to the mark with methanol. Final concentration is 250 ng/ml.

7.2 Gas chromatographic conditions

Injector temperature: 270° C

Detector temperature: 300° C

Column temperature ramp: 50° C/1 min to 150° C at 20° C/min, hold for 0 mins;

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150° C to 200° C @ 5° C/min, hold for 0 mins; 200° C to 280° C @ 25° C/min, hold for 11 min.

Gases: Carrier Gas - Helium, ultra pure carrier, flow of 1 to 2 ml per minute

Make up Gas - 5% Methane/Argon, flow of 40 to 50 ml per minute

7.3 Instrument Calibration

7.3.1 Set-up the GC according to the analytical conditions specified in Section 7.2 and load the samples onto the autosampler.

7.3.2 Use the external calibration technique to calibrate the GC system.

7.3.3 Retention windows will be determined using the following procedure which is based on the 72 hour calibration procedure defined in SW-846 method 8000.

7.3.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

7.3.3.2 Calculate the standard deviation of the three absolute retention times for each single component standard. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

7.3.3.2.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.3.3.2.2 In those cases where the standard deviation for a particular standard is zero, the analyst must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

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- 7.3.3.3 The analyst must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The retention time data must be retained.
- 7.3.3.4 An absolute retention time for each analyte is determined daily by calculating the average retention time for that analyte for the calibration curve standards and all of the continuing calibration standards. This retention time \pm the retention window calculated in section 9.2.1-9.2.2 will be used by the analyst to qualitatively identify the peak.
- 7.3.3.5 If an analyte retention time is outside of the calculated window documentation must be provided in order to justify the qualitative identification of the peak.
- 7.3.4 Continuing Calibration Check Standard (CCS) - A CCS must be run by the analyst at a minimum interval of every 10 samples and at the end of the run. The response of the CCS should be within 15 percent of the same standard in the calibration curve. If the continuing calibration fails the requirements, the analyst must follow the corrective actions outlined in the QA/QC Manual before continuing with the analysis.
- 7.3.5 The analyst must check the response of the target analytes within each sample. If the response of an analyte exceeds the calibration range for that analyte the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL

For each lot of samples, extract and analyze the following required QC:

- 5% Method Blank
- 5% Standard Matrix Spike
- 5% Sample Matrix Spike (if required by the client)
- 5% Sample Matrix Spike Duplicate (if required by the client)
- 100% samples spiked with surrogate

Note: The number of QC required is the actual number of QC samples rounded up to the nearest whole number, i.e, 5% = 1 QC for 1-20 samples; 2 QC for 21-40 samples, etc.

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9.0 CALCULATIONS

9.1 Target responses are transferred to the Laboratory Data Management System, CLASS (Chemical Laboratory Analytical and Scheduling System), along with any relevant sample information. The concentration is calculated using the regression equation calculated by CLASS. Final samples results are corrected for sample volume or weight, extract volume, percent moisture for solid samples, dilution factors and any applicable conversion factors.

9.2 Peak Identification

Analyte response that lies within the established retention time will be considered to be tentatively identified. Analyst must use their judgement as to whether the peak may represent a target compound by examining such factors as peak shape, resolution from interferences and matrix "noise".

9.3 Confirmation

Analytes that are tentatively identified on the primary column must be confirmed by analysis on a different column with a different liquid phase. In order to confirm an analyte a response must be present in the retention windows for the analyte on both the primary column and the confirmation column. The retention windows will be calculated the same way for both columns. Decision points to be made for the identification and reporting of a target analyte are:

9.3.1 Is there a response in the retention window of a target analyte on the primary column and the sum of the responses are above the reporting limit (RL)?

No. No further action is necessary and the analyte is reported as <RL.

Yes. Analyze the sample extract on the confirmation column.

9.3.2 Is there a response on the confirmation column in the retention window of the target analyte and the sum of the responses are above the criterion

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of detection?

No. The analyte is not confirmed and the analyte is reported as <RL adjusted for any dilutions required.

Yes. Determine ability to identify peak.

9.3.3 Is the peak well defined?

Yes. The analyte is confirmed and the response of the target analyte is reported from the primary column analysis.

No. There is considerable interference on the confirmation column analysis which in the analyst's judgement precludes their ability to identify a peak in the retention window of interest. The analyte is considered as not confirmable. The analyte will be reported with the concentration calculated from the primary column and flagged with a "Q".

Definition: COD = one half of the detection limit

9.4 Analyte Quantitation

Peak areas or heights may be used to calculate analyte concentrations. The same technique must be used for both the standard curve and the samples analyte.

10.0 SAFETY PRACTICES

The analyst must be aware of the hazards associated with the chemicals used in this method. The hazards are minimized by reducing the possibility of accidental absorption or ingestion. Eating and drinking are not permitted in areas where chemicals are used or stored. Lab coats, gloves and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals used, the Material Safety Data Sheets (MSDS) must be consulted.

The target compounds in this method are toxic. The preparation of all standards should be performed in a laboratory hood. Adequate dermal and eye protection must be used when handling samples and standards.

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Ether, hexane, and acetone are volatile, flammable liquids, and should not be used around an open flame or source of spark. Use flammable solvents only in properly vented areas.

Methylene chloride is an irritant and central nervous system depressant. In addition, it is a suspect carcinogen and should be used only in well ventilated areas.

11.0 REFERENCE

- 11.1 EPA Method 8150 - Test Methods for Evaluating Solid Wastes, EPA SW 846 3rd Edition, September 1986.
- 11.2 EPA Method 615 - Method for Organic Chemical Analyses of Municipal and Industrial Wastewater, EPA 600/4-82-057.
- 11.3 EPA Method 8150B - Test Methods for Evaluating Solid Wastes, EPA SW 846 3rd Edition (Revision 2), November 1992.

12.0 ATTACHMENTS

- 12.1 ATTACHMENT A - Method Reporting and Detection Limits Summary Table

ATTACHMENT A

WATER

ANALYTE	METHOD DETECTION LIMIT (MDL) ug/L	REPORTING LIMIT (RL) ug/L
Dalapon	0.018	0.126
Dicamba	0.032	0.126
MCPPP	0.41	3.0
MCPA	2.6	3.0
Dichloroprop	0.078	0.126
2,4-D	0.030	0.126
Silvex	0.013	0.126
2,4,5-T	0.013	0.126
Dinoseb	0.015	0.126
2,4-DB	0.053	0.126

SOIL

ANALYTE	METHOD DETECTION LIMIT (MDL) ug/Kg	REPORTING LIMIT (RL) ug/Kg
Dalapon	1.1	20
Dicamba	1.3	20
MCPPP	33	400
MCPA	180	400
Dichloroprop	2.3	20
2,4-D	1.8	20
Silvex	0.8	20
2,4,5-T	0.9	20
Dinoseb	1.8	20
2,4-DB	4.1	20

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

**TITLE: CATION-EXCHANGE CAPACITY OF SOILS (AMMONIUM ACETATE)
(EPA METHOD 9080A)**

Effective Date: _____

Prepared By: Kathleen K. Allen _____

Reviewed By: Kenneth U. Erundu _____

Approved By: John J. Mousa
(Gainesville Laboratory Director) _____

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**TITLE: CATION-EXCHANGE CAPACITY OF SOILS (AMMONIUM ACETATE)
(EPA METHOD 9080A)**

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) is used to determine the cation-exchange capacity of soils. This sop is not applicable to soils containing appreciable amounts of vermiculite clays, kaolin, halloysite, or other 1:1-type clay minerals. They should be analyzed by the sodium acetate method (Method 9081). Method 9081 is also generally the preferred method for very calcareous soils.

2.0 SUMMARY

A soil is mixed with an excess of 1 N ammonium acetate solution. This results in an exchange of the ammonium cations for exchangeable cations present in the soil. The excess ammonium is removed, and the amount of exchangeable ammonium is determined.

3.0 INTERFERENCES

- 3.1 Soils containing appreciable vermiculite clays, kaolin, halloysite, or other 1:1-type clay minerals will often give lower values for exchange capacity.
- 3.2 With calcareous soils, the release of calcium carbonate from the soil into the ammonium acetate solution limits the saturation of exchange sites by the ammonium ion. This results in artificially low cation-exchange capacities.

4.0 APPARATUS AND MATERIALS

- 4.1 Erlenmeyer flask: 500-mL.
- 4.2 Buchner funnel or equivalent: 55-mm.
- 4.3 Sieve: 2-mm.
- 4.4 Agate mortar.
- 4.5 Analytical balance: capable of weighing to 0.01 g.

5.0 REAGENTS

- 5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first

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ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 5.2 Reagent water.
- 5.3 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with reagent water to a volume of approximately 1 liter. Add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH , as needed, to obtain a pH of 7. Dilute the solution to the volume of 2 liters with water.
- 5.4 Isopropyl alcohol: 99%.
- 5.5 Ammonium chloride (NH_4Cl), 1 N: Dissolve 53.49 g of NH_4Cl in reagent water, adjust the pH to 7.0 with NH_4OH , and dilute to 1 L.
- 5.6 Ammonium chloride (NH_4Cl), 0.25 N: dissolve 13.37 g of NH_4Cl in reagent water, adjust the pH to 7.0 with NH_4OH , and dilute to 1 L.
- 5.7 Ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$), 10%: Add 90 mL of reagent water to 10 g of ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) and mix well.
- 5.8 Dilute ammonium hydroxide (NH_4OH): Add 1 volume of concentrated NH_4OH to an equal volume of water.
- 5.9 Silver nitrate (AgNO_3), 0.10 N: Dissolve 15.39 g of AgNO_3 in reagent water, mix well, and dilute to 1 L.
- 5.10 Reagents for distillation option:
 - 5.10.1 Sodium chloride, NaCl (acidified), 10%: Dissolve 100 g of NaCl (ammonium-free) in 900 mL of reagent water; mix well. Add approximately 0.42 mL of concentrated HCl to make the solution approximately 0.005 N.
 - 5.10.2 Sodium hydroxide (NaOH), 1 N: Dissolve 40 g of NaOH in reagent water and dilute to 1 L.
 - 5.10.3 Boric acid (H_3BO_3), 2% solution: Dissolve 20 g H_3BO_3 in 980 mL reagent water and mix well.

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- 5.10.4 Standard sulfuric acid (H_2SO_4), 0.1 N. Add 2.8 mL of concentrated sulfuric acid to reagent water and dilute to 1 L. Standardize against a base of known concentration or purchase a certified commercially prepared 0.1N H_2SO_4 and standardize.
- 5.10.5 Bromocresol green-methyl red mixed indicator: Crush 0.1 g of bromocresol green and 2 mL 0.1 N NaOH in an agate mortar. Add 95% ethyl alcohol to obtain a total volume of 100 mL. Crush 0.1 g of methyl red with a few mL of 95% ethyl alcohol in an agate mortar. Add 3 mL of 0.1 N NaOH and dilute the solution to a volume of 100 mL with 95% ethyl alcohol. Mix 75 mL of the bromocresol green solution with 25 mL of the methyl red solution. Dilute the mixture to 200 mL with 95% ethyl alcohol.

6.0 PRECEDURE

- 6.1 Sieve a sample aliquot of the soil through a 2-mm screen and allow the sieved soil to air dry (at a temperature of $\leq 60^\circ\text{C}$). Place 10 g of the air-dried soil in a 500-mL Erlenmeyer flask and add 250 mL of neutral, 1 N NH_4OAc , (Section 5.3). (Use 25 g of soil if the exchange capacity is very low, e.g., 3-5 meq per 100 g.) Shake the flask thoroughly and allow it to stand overnight.
- 6.2 Filter the soil with light suction using a 55-mm Buchner funnel or equivalent. Do not allow the soil to become dry and cracked.
- 6.3 Rinse the soil with the neutral NH_4OAc reagent the test for calcium in the effluent solution is negative.
- 6.3.1 T.D. test for calcium, add a few drops of 1 N NH_4Cl and 10% ammonium oxalate, each to effluent. Dilute with NH_4OH to 10 mL and heat the solution to near the boiling point. The presence of calcium is indicated by a white precipitate or turbidity.)
- 6.4 Rinse the soil four times with neutral 1 N NH_4Cl and once with 0.25 N NH_4Cl .
- 6.5 Rinse the soil with 150 to 200 mL of 99% isopropyl alcohol. Test the leachate for chloride use 0.10 AgNO_3 . When the test for chloride is negative allow the soil to drain thoroughly.
- 6.6 Determine the adsorbed NH_4 or by the acid-NaCl method (Section 6.8).

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6.7 Acid-NaCl method:

- 6.7.1 Rinse the ammonium-saturated soil with 10% acidified NaCl until 225 mL have passed through the sample. Add small portions at a time, allowing each portion to pass through the sample before adding the next portion.
- 6.7.2 Transfer the leachate quantitatively to an 800-mL Kjeldahl flask, add 25 mL of 1 N NaOH. Distill 60 mL of the solution into 50 mL of 2% H_3BO_3 .
- 6.7.3 Add 10 drops of bromocresol green-methyl red mixed indicator to the distillate. Titrate the distillate with standard 0.1 N H_2SO_4 . The color change is from bluish green through bluish purple to pink at the end point. Run blanks on the reagents. Correct the titration figure for the blanks and calculate the milliequivalents of ammonium in 100 g of soil.
- 6.7.4 Results should be reported as "determined with ammonium acetate" at pH 7.

7.0 QUALITY CONTROL

- 7.1 Method blanks (MB) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. MB consists of deionized (DI) water.
- 7.2 Replicate samples must be analyzed at a frequency of 5% for every analytical batch unless, specified differently by a project.

Note: The actual number of QC required is rounded up to the nearest whole number, i.e., 5% = 1 QC for 1-20 samples; 2 QC for 21-40 samples, etc.

8.0 REFERENCES

Test Methods for Evaluating Solid Waste (EPA 9080A), SW-846, Third Edition, November 1990.

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

TITLE: TOTAL ORGANIC CARBON (TOC) IN WATER (EPA METHOD 415.1/9060 MODIFIED)

Effective Date: 2/3 3/2/95
was 1/1/95

Prepared by: Kathleen K. Allen

Reviewed by: Kenneth U. Erondü

Approved by: John J. Mousa
(Gainesville Laboratory Director)

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TITLE: TOTAL ORGANIC CARBON (TOC) IN WATER (EPA METHOD 415.1/9060 MODIFIED)

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a consistent method for the determination of total organic carbon in water samples.

2.0 SCOPE

2.1 This method includes the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes.

2.2 Detection limit for this method is 1.0 mg/L.

2.3 This procedure is applicable only to homogeneous samples which can be drawn into the apparatus reproducible by means of the autosampler.

3.0 SUMMARY OF METHOD

Organic carbon in a sample is converted to carbon dioxide (CO₂) by catalytic combustion. The CO₂ formed is measured directly by a non-dispersive infrared detector. The amount of CO₂ is directly proportional to the concentration of carbonaceous material in the sample.

4.0 METHOD INTERFERENCES

Removal of carbonate and bicarbonate by acidification and purging with purified gas will result in the loss of volatile organic substances.

5.0 APPARATUS AND MATERIALS

5.1 Dohrmann DC-190 High Temperature Total Organic Carbon (TOC) Analyzer with autosampler.

5.2 8 mL autosampler vials.

5.3 100 mL volumetric glassware.

5.4 Adjustable eppendorf or syringe, calibrated each day of use.

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6.0 REAGENTS

- 6.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.
- 6.2 Potassium Hydrogen Phthalate, Stock Standard, 2000 mg carbon/L: Dissolve 0.4256 g of potassium hydrogen phthalate (primary standard grade) in distilled water and dilute to 100.0 mL. This solution expires in thirty days.
- 6.3 Potassium Hydrogen Phthalate, Control Stock Standard, 2000 mg carbon/L: Dissolve 0.4256 g of potassium hydrogen phthalate (primary standard grade) in distilled water and dilute to 100.0 mL. This solution expires in thirty days.

NOTE: It is recommended that these chemicals be obtained from a different supplier than the chemicals used to make the Stock Standard (Section 6.2). However, if the chemicals are obtained from the same manufacturer, they must be from different lots than the chemicals used to prepare the Stock Standard.

- 6.4 Preparation of Calibration Standards: Prepare a series of standards by pipeting the appropriate volumes of the 2000 mg carbon/L stock standard (Section 6.2), into a series of 100 mL volumetric flasks and dilute to volume with distilled water. It is recommended that the following volumes of the 2000 mg carbon/L stock standard be used to obtain a working curve of approximately 1 - 100 mg/L.

<u>Volume of 2000 mg/L Stock Standard used (mL)</u>	<u>Concentration (mg/L)</u>
0.05	1.0
0.125	2.5
0.25	5.0
0.5	10.0
1.5	30.0
5.0	100.0

These standards are to be made fresh daily.

- 6.5 Intermediate Control Stock Standard: Prepare a 20 mg carbon/L intermediate control stock by pipetting 1 mL of Control Stock Standard (Section 6.3) into a 100 mL volumetric flask. Bring to volume with distilled water. This solution is to be made fresh daily.

7.0 SAFETY PRECAUTIONS

The analyst must be aware of the hazards associated with the chemicals used in this method. The hazards are minimized by reducing the possibility of accidental absorption or ingestion. Eating and drinking are not permitted in areas where chemicals are used or stored. Lab coats, gloves and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals used, the Material Safety Data Sheets (MSDS) must be consulted.

8.0 PROCEDURE

- 8.1 Analyze the calibration standards in order of increasing concentration. All standard concentrations should be within $\pm 20\%$ of stated concentration. If the standards are outside of this criteria, the instrument must be re-calibrated. To calibrate the instrument, analyze one standard in duplicate, preferably the midpoint standard (and follow by analyzing distilled water). Press the calibration button and re-analyze all calibration standards to ensure that the instrument is properly calibrated.
- 8.2 All samples (be sure to use the 'S' fraction), standards and QC aliquots should be run in duplicate with the instrument set to print averages for total carbon, inorganic carbon and organic carbon. If the sample response is outside the standard curve, the sample must be diluted and reanalyzed.
- 8.3 Any reading resulting in a "Time Out Error" will not be used to determine the sample concentration. The sample must be reanalyzed.

9.0 CALCULATION

A calibration curve is prepared by plotting each standard response against concentration values. The sample concentration is calculated by plotting the average TOC sample responses against the standard curve. The sample concentration from the curve must be multiplied by any dilution factors.

10.0 QUALITY CONTROL

- 10.1 Method blanks (MB) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. The MB consists of distilled water.
- 10.2 Standard Spike (SP) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. Use the 20 mg carbon/L Intermediate Control Stock Standard (Section 6.5) as the SP.

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- 10.3 Sample Matrix Spike (SPM) and Sample Matrix Spike Duplicate (SPMD) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. SPM and SPMD are prepared by making a 1:1 dilution of the sample and the 20 mg carbon/L Intermediate Control Stock Standard.

NOTE: The actual number of QC required is rounded up to the nearest whole number, i.e., 5% = 1 QC for 1-20 samples; 2 QC for 21-40 samples, etc.

- 10.4 The Continuing Calibration Verification (CCV) is a quality control check used to ensure the validity of the curve while running samples. The CCV is the re-analysis of one of the original calibration standards. It is recommended that the midpoint standard be used.

11.0 REFERENCES

- 11.1 Methods for Chemical Analysis of Water and Wastes, (EPA method 415.1) EPA-600/4-79-020, Revised March 1983.
- 11.2 Test Methods for Evaluating Solid Waste, (EPA Method 9060) EPA SW-846, 3rd Edition, September 1990.

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

**TITLE: TOTAL ORGANIC CARBON (TOC) IN SOIL (MODIFIED EPA
METHOD '9060)**

Effective Date: 10/28/95

Prepared by: Kathleen K. Allen

Reviewed by: Kenneth U. Erundu

Approved by: John J. Mousa
(Gainesville Laboratory Director)

[Signature] 9/28/95
[Signature] 4/28/95
John J. Mousa 9/28/95
[Signature]

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TITLE: TOTAL ORGANIC CARBON (TOC) IN SOIL (MODIFIED EPA METHOD 9060)

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a consistent method for the determination of total organic carbon in soil and sediment samples.

2.0 SCOPE

2.1 This method includes the measurement of organic carbon in soil and sediment samples.

2.2 Detection limit for this method is 360 mg/Kg.

2.3 This procedure is applicable only to homogeneous samples.

3.0 SUMMARY OF METHOD

Any inorganic carbon present in the sample is driven off by acidification within the instrument. Organic carbon in the sample is converted to carbon dioxide (CO₂) by catalytic combustion. The CO₂ formed is measured directly by a non-dispersive infrared detector. The amount of CO₂ is directly proportional to the concentration of carbonaceous material in the sample.

4.0 METHOD INTERFERENCES

Removal of carbonate and bicarbonate by acidification will result in the loss of volatile organic substances. Any inconsistency in the sample matrix will give vastly different responses due to the small mass used (approximately 50 mg) in the analysis.

5.0 APPARATUS AND MATERIALS

5.1 Dohrmann DC-190 High Temperature Total Organic Carbon (TOC) Analyzer with soil furnace unit.

5.2 Platinum sample boats.

5.3 Analytical balance capable of weighing to 0.1 mg.

5.4 Syringe, 50 µl.

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6.0 REAGENTS

- 6.1 Distilled water used in the preparation of standards should be ultra pure to reduce the carbon concentration of the diluent.
- 6.2 Potassium Hydrogen Phthalate, Stock Standard, 2000 mg carbon/L: Dissolve 0.4256 g of potassium hydrogen phthalate (primary standard grade) in distilled water and dilute to 100.0 mL. This solution expires in thirty days.
- 6.3 Potassium Hydrogen Phthalate, Control Stock Standard, 2000 mg carbon/L: Dissolve 0.4256 g of potassium hydrogen phthalate (primary standard grade) in distilled water and dilute to 100.0 mL. This solution expires in thirty days.
- 6.4 Potassium Hydrogen Phthalate, Stock Standard, 10,000 mg carbon/L: Dissolve 2.1264 g of potassium hydrogen phthalate (primary standard grade) in distilled water and dilute to 100.0 mL. This solution expires in thirty days.

NOTE: It is recommended that these chemicals be obtained from a different supplier than the chemicals used to make the Stock Standard (Section 6.2). However, if the chemicals are obtained from the same manufacturer, they must be from different lots than the chemicals used to prepare the Stock Standard.

- 6.5 Phosphoric Acid, 85%.

7.0 SAFETY PRECAUTIONS

- 7.1 The analyst must be aware of the hazards associated with the chemicals used in this method. The hazards are minimized by reducing the possibility of accidental absorption or ingestion. Eating and drinking are not permitted in areas where chemicals are used or stored. Lab coats, gloves and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals used, the Material Safety Data Sheets (MSDS) must be consulted.
- 7.2 Phosphoric acid is corrosive and adequate eye and dermal protection are required.

8.0 PROCEDURE

- 8.1 Turn on the main power to the boat sampler module, boat gas, gas carrier and the switch gas line to up position. Purge line must be hooked to soil combustion unit. Wait for green temperature light on boat sampling module to come on.

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- 8.2 Set the sample mass to 20 mg and inject 20 μ l of the 10,000 mg/L Stock Standard (Section 6.4) onto the platinum sample boat and analyze. This response is used to calibrate the instrument and corresponds to a 10,000 mg/Kg sample. The concentration of this standard should be within $\pm 10\%$ of the true value. If the 10,000 mg/Kg standard is outside the acceptance criteria, the instrument must be re-calibrated. To calibrate the instrument, analyze the 10,000 mg/Kg Stock Standard and press the calibration button. An injection of 3.0 μ l of the 2000 mg/L Stock Standard (Section 6.2) is performed to verify that the detection limit is achievable. An injection of 10 μ l of the 2000 mg/L Stock Standard (Section 6.2) is performed and it corresponds to a 1000 mg/Kg sample.

NOTE: The instrument manufacturer states that a one point calibration is all that is needed with the DC-190. The instrument is linear and a Time Out Error will be given if the capacity of the instrument is exceeded.

- 8.3 The sample mass must be entered into the instrument prior to analysis or the instrument will not integrate the carbon counts. Sample mass used to perform the analysis should be as small as can be accurately weighed.
- 8.4 All samples should be run in duplicate with the average of at least two injections being reported as the concentration of the sample.
- 8.5 A reading that results in a "Time Out Error" (TOE) can not be used for reporting total organic carbon. The sample should be reanalyzed using a smaller mass. If it is not possible to get a smaller mass that can be accurately weighed into the sample boat, the TOE can be reported but the Lab Coordinator should be informed by noting the instrument TOE reading in the batch.

9.0 CALCULATION

- 9.1 The zero, 300, 1000 and 10,000 mg/kg standards are entered into CLASSTM to show linearity but a calculation technique of FINAL is used. The DC 190 Carbon Analyzer provides a printout showing all injections and final concentrations of TOC in mg/Kg. This strip chart is to be included in the data batch along with a copy of the instrument run log.
- 9.2 To have CLASSTM perform the calculation, use the store list AVG and the AVG calculation technique. Enter each response for a given sample and CLASSTM will average the values (this calculation technique will average up to 4 injections per sample).

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10.0 QUALITY CONTROL

- 10.1 Method blanks (MB) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. The MB consists of an empty boat with the sample mass set to 20 mg.
- 10.2 Standard Spike (SP) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. An injection of 10.0 μ l of the Control Stock Standard (Section 6.3) is injected onto an empty boat (with the sample mass setting of 20 mg). The target is 1000 mg/Kg.
- 10.3 Sample Matrix Spike (SPM) and Sample Matrix Spike Duplicate (SPMD) must be analyzed at a frequency of 5% for every analytical batch, unless specified differently by a project. SPM and SPMD are prepared by adding 10.0 μ l of the Control Stock Standard (Section 6.3) to a pre-weighed sample. The target is calculated as follows:

$$20,000/\text{mg sample used} = \text{mg/Kg target}$$

NOTE: The actual number of QC required is rounded up to the nearest whole number, i.e., 5% = 1 QC for 1-20 samples; 2 QC for 21-40 samples, etc.

- 10.4 The Continuing Calibration Verification (CCV) is a quality control check used to ensure the validity of the curve while running samples. The CCV is the re-analysis of 10.0 μ l of the Stock Standard (Section 6.2) with the sample mass set to 20 mg. This results in a target of 1000 mg/Kg. A CCV should be run every twenty injections and must be within $\pm 15\%$ of the initial standard response.
- 10.5 The Continuing Calibration Blank (CCB) is a quality control check used to demonstrate the stability of the baseline. The CCB is the same as the analysis of a method blank and should be run every 20 injections.

11.0 REFERENCES

- 11.1 DC-190 High-Temperature TOC Analyzer Operation Manual, Rosemount Analytical, Inc. May 1991.
- 11.2 Test Methods for Evaluating Solid Waste, (EPA Method 9060) EPA-SW-846, 3rd Edition, September 1986.

Appendix B

EXAMPLE FIELD AUDIT CHECKLIST

FIELD CHECKLIST

Signature of Auditor _____ Date of Audit _____

Project Coordinator _____ Project No. _____

Project Location _____

Type of Investigation _____
(Authority, Agency)

Briefing with Project Coordinator

Yes _ No _ N/A _ 1. Was a project plan prepared? If yes, what items are addressed in the plan?

Yes _ No _ N/A _ 2. Were additional instructions given to project participants (i.e., changes in project plan)? If yes, describe these changes.

Yes _ No _ N/A _ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

Yes _ No _ N/A _ 4. Is there a map of sampling locations? If yes, where is the map?

_____

Yes ☐ No ☐ N/A ☐

5. Do the investigators follow a system of accountable documents? If yes, what documents are accountable?

Yes ☐ No ☐ N/A ☐

6. Is there a list of accountable field documents checked out to the project coordinator? If yes, who checked them out and where is this documented?

Yes ☐ No ☐ N/A ☐

7. Is the transfer of field documents (sample tags, chain-of-custody records, logbooks, etc.) from the project coordinator to the field participants documented? If yes, where is the transfer documented?

Yes ☐ No ☐ N/A ☐

8. Have the team members received the adequate training for their position? Documented?

Yes ☐ No ☐ N/A ☐

9. Have the team members received the required number of hours of OSHA training.



FIELD CHECKLIST

FIELD OBSERVATIONS

Yes ☐ No ☐ N/A ☐ 1. Was permission granted to enter and inspect the facility (required if RCRA inspection)?

Yes ☐ No ☐ N/A ☐ 2. Is permission to enter the facility documented? If yes, where is it documented?

Yes ☐ No ☐ N/A ☐ 3. Were split samples offered to the facility. If yes, was the offer accepted or declined?

Yes ☐ No ☐ N/A ☐ 4. Is the offering of split samples recorded? If yes, where is it recorded?

Yes ☐ No ☐ N/A ☐ 5. If the offer to split samples was accepted, were the split samples collected? If yes, how were they identified?



Yes ☐ No ☐ N/A ☐

6. Are the number, frequency and types of field measurements, and observations taken as specified in the project plan or as directed by the project coordinator? If yes, where are they recorded?

Yes ☐ No ☐ N/A ☐

7. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers were used?

Yes ☐ No ☐ N/A ☐

8. Are samples preserved as required? If no or N/A, explain.

Yes ☐ No ☐ N/A ☐

9. Are the number, frequency, and types of samples collected as specified in the project plan or as directed by the project coordinator? If no, explain why not?

Yes ☐ No ☐ N/A ☐

10. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.



Yes ☐ No ☐ N/A ☐ 11. Is sample custody maintained at all times? How?

Yes ☐ No ☐ N/A ☐ 12. Is the following information completed on each chain-of-custody record?

- Sample identification number;
- Sample collector's signature;
- Date and time of collection;
- Place and address of collection;
- Waste sample description;
- Shipper's name and address;
- Name and address of organization(s) receiving sample;
- Signatures and titles of persons involved in chain-of-possession; and
- Inclusive dates of possession for each possession.

Yes ☐ No ☐ N/A ☐ 13. Does a sample analysis sheet accompany all samples on delivery to the laboratory sample custodian?

Yes ☐ No ☐ N/A ☐ 14. At the minimum, has the following information been completed on each sample analysis request sheet?

- Name of person receiving sample (sample custodian);
- Laboratory sample number;
- Date of sample receipt;
- Sample allocation;
- Analyses to be performed;
- Collector's name, affiliation name, address, and phone number;
- Date and time of sampling;



- Location of sampling; and
- Special handling and/or storage requirements.

Yes ☐ No ☐ N/A ☐ 15. Has a field custodian been assigned for sample recovery, preservation, and storage until shipment?

Yes ☐ No ☐ N/A ☐ 16. Where applicable, are sample collection containers rinsed three times with the sample material prior to collection?



Yes ☐ No ☐ N/A ☐ 17. Are glass containers with Teflon-lined screw caps used to collect the following types of samples?

- Water samples for organic analyses?
- Soil and sediment samples?
- Liquid and solid hazardous waste samples (*)?

Yes ☐ No ☐ N/A ☐ 18. Are polyethylene bottles with solid polyethylene-lined caps used to collect the following types of samples?

- Water samples for metal analysis?
- Water samples for pH and fluoride analysis?
- Water samples for cyanide analysis?

Yes ☐ No ☐ N/A ☐ 19. Are amber glass or aluminum foil-wrapped glass bottles used for samples suspected of being photosensitive?

* Highly alkaline wastes and wastes known to contain hydrofluoric acid should be collected in plastic containers. If it is suspected that highly alkaline materials or hydrofluoric acid is present, a small sample should be tested to determine if it reacts with the sample container.



QUALITY ASSURANCE/QUALITY CONTROL
SAMPLE DOCUMENTATION AND CHAIN-OF-CUSTODY

Yes ☐ No ☐ N/A ☐ 1. Is the following information being recorded in the field log book or on data sheets?

- Project name and project number;
- Purpose of sampling (e.g., quarterly sampling, resample to confirm previous analysis, initial site assessment, etc.);
- Date and time each sample was collected;
- Date and starting/stopping times (Hr:Min) for air samples;
- Date and well bailing time for groundwater;
- Blank, duplicate and split sample identification numbers;
- Sample description including type (i.e., soil, sludge, groundwater, etc.);
- Field measurement results (i.e., conductivity, pH, dissolved oxygen, combustible gas (e.g., LEL), radioactivity, etc.);
- Preservation method for each sample;
- Type and quantity of containers used for each sample;
- Weather conditions at time of sampling;
- Photographic log identifying subject, reason for photograph, date, time, direction in which photograph was taken, number of the picture on the roll;
- Sample destination;
- Analyses to be performed on each sample;
- Reference number from all forms on which the sample is listed or labels attached to the sample (i.e., chain-of-custody, bill of lading or manifest forms, etc.);
- Name(s) of sampling personnel; and



- Signature of person(s) making entries on each page.

Yes _ No _ N/A _

2. Is a chain-of-custody record completed for all samples collected?



CHECKLIST FOR MECHANICALLY CORED SAMPLES

Yes ☐ No ☐ N/A ☐ 1. Was the rig set up at a staked and cleared borehole location?

Yes ☐ No ☐ N/A ☐ 2. Was the location, date, time, and other pertinent information recorded on boring log form?

Yes ☐ No ☐ N/A ☐ 3. Was polybutyrate core tubes cut to specification and placed into core barrel?

Yes ☐ No ☐ N/A ☐ 4. Was auguring and coring conducted according to the following sequence: 0-1 ft, 1-4 ft, 4-5 ft, 5-9 ft, and 9-10 ft, etc.?

Yes ☐ No ☐ N/A ☐ 5. Was the core barrel removed from the borehole and opened at the completion of each coring interval?



Yes ☐ No ☐ N/A ☐

6. Was the 12-inch sections for laboratory analysis removed, capped with Teflon film lined plastic caps, sealed with tape, and immediately placed in a cooler?

Yes ☐ No ☐ N/A ☐

7. Were core sections which were previously etched length-wise taped with plastic caps to prevent opening during transport to the support facility?

Yes ☐ No ☐ N/A ☐

8. Were the polybutyrate line sections marked with an arrow to the top end, the boring number, and depth interval? Was a label giving the same information as well as the project name, number, the date, and the sampler's initials attached to the core in the sample handling trailer or at the site?

Yes ☐ No ☐ N/A ☐

9. Were clean polybutyrate liners placed in a clean core barrel for each additional coring increment to be drilled?

Yes ☐ No ☐ N/A ☐

10. Did the boring reach a predetermined depth or encounter the water table, whichever came first?



Yes ☐ No ☐ N/A ☐

11. For trench disposal areas was the coring performed to the maximum depth of observable contamination?

Yes ☐ No ☐ N/A ☐

12. Were all core sections transported to the support facility for logging and sample shipment preparation?

Yes ☐ No ☐ N/A ☐

13. Was the boring stake left in the ground adjacent to the borehole and a board placed over the hole until it was grouted?

Yes ☐ No ☐ N/A ☐

14. Were all boreholes greater than 1 ft in depth grouted the same day of construction and the borehole location stake placed in the grout?

Yes ☐ No ☐ N/A ☐

15. Were one foot deep borings backfilled with native materials available adjacent to the boring?



Yes ☐ No ☐ N/A ☐

16. Were the augers, and other downhole equipment decontaminated in the field prior to moving to the next borehole location upon completion of each boring?

Yes ☐ No ☐ N/A ☐

17. When all borings in a specific source were completed was the drill rig initially cleaned at the source location?

Yes ☐ No ☐ N/A ☐

18. Upon completion of the initial cleaning was the drill rig transported to the decontamination pad where it was thoroughly steam-cleaned before entering another source area?

Yes ☐ No ☐ N/A ☐

19. Were enough augers and core barrels available so that when one set was in use a second set was being decontaminated?

Yes ☐ No ☐ N/A ☐

20. At the end of the working day did all equipment, except the drill rig, and personnel proceed to the decontamination pad where decontamination procedures were initiated?



Yes ☐ No ☐ N/A ☐

21. Were all bore cuttings drummed and stored while awaiting USAEC's directions for disposal?



CHECKLIST FOR HAND CORED SAMPLES

Yes ☐ No ☐ N/A ☐

1. Was a piece of Teflon film and plywood placed over the top of the polybutyrate tube and the tube pushed or driven into the ground by hand?

Yes ☐ No ☐ N/A ☐

2. Was the tube removed from the ground by shovel, the tube exterior wiped clean, the ends capped with Teflon film lined plastic caps, and sealed with tape?

Yes ☐ No ☐ N/A ☐

3. Were the sample tubes marked with the boring number, the depth of the interval sampled, and the upward direction?

Yes ☐ No ☐ N/A ☐

4. Was a label containing the same information written on the sample tube as well as the project name, number, the date, and sampler's initials taped to the outside of the core?

Yes ☐ No ☐ N/A ☐

5. Were cores logged and stored in a cooler with commercially available Blue Ice prior to and during transport to the support facility sampling area where they were logged for shipment?



FIELD CHECKLIST
DOCUMENT CONTROL

Yes ☐ No ☐ N/A ☐

1. Have all unused and voided accountable documents been returned to the coordinator by the team members?

Yes ☐ No ☐ N/A ☐

2. Were any accountable documents lost or destroyed? If yes, have document numbers of all lost or destroyed accountable documents been recorded and where are they recorded?

Yes ☐ No ☐ N/A ☐

3. Are all samples identified with sample tags? If no, how are samples identified?

Yes ☐ No ☐ N/A ☐

4. Are all sample tags completed (e.g., station number, location, date, time, analyses, signatures of samplers, type, preservatives, etc.)? If yes, describe types of information recorded.



Yes ☐ No ☐ N/A ☐

5. Are all samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

Yes ☐ No ☐ N/A ☐

6. If used, are the sample tag numbers recorded on the chain-of-custody documents?

Yes ☐ No ☐ N/A ☐

7. Does information on sample tags and chain-of-custody records match?

Yes ☐ No ☐ N/A ☐

8. Does the chain-of-custody record indicate the method of sample shipment?

Yes ☐ No ☐ N/A ☐

9. Is the chain-of-custody record included with the samples in the shipping container?

Yes ☐ No ☐ N/A ☐

10. If used, do the sample traffic reports agree with the sample tags?



Yes ☐ No ☐ N/A ☐

11. If required, has a receipt for samples been provided to the facility (required by RCRA)? Describe where offer or a receipt is documented.

Yes ☐ No ☐ N/A ☐

12. If used, are blank samples identified?

Yes ☐ No ☐ N/A ☐

13. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

Yes ☐ No ☐ N/A ☐

14. If used, are spiked samples identified?

Yes ☐ No ☐ N/A ☐

15. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

Yes ☐ No ☐ N/A ☐

16. Are logbooks dated upon receipt from the project coordinator?



Yes ☐ No ☐ N/A ☐

17. Are logbooks project-specific (by logbook or by page)?

Yes ☐ No ☐ N/A ☐

18. Are logbook entries dated and identified by author?

Yes ☐ No ☐ N/A ☐

19. Is the facility's approval or disapproval to take photographs noted in a logbook?

Yes ☐ No ☐ N/A ☐

20. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes ☐ No ☐ N/A ☐

21. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes ☐ No ☐ N/A ☐

22. Are sample tag numbers recorded? If yes, describe where they are recorded.



FIELD CHECKLIST

DEBRIEFING WITH PROJECT COORDINATOR

Yes ☐ No ☐ N/A ☐

1. Was a debriefing held with project coordinator and/or other participants?

Yes ☐ No ☐ N/A ☐

2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.

Yes ☐ No ☐ N/A ☐

3. Was a copy of the field checklist left with the project coordinator at the conclusion of the debriefing?



Appendix C
QUALITY CONTROL CRITERIA

Table C1: Quality Control Performance Criteria for Matrix Spikes/Matrix Spike Duplicates, Laboratory Control Samples, and Surrogates
U.S. Environmental Protection Agency Contract Laboratory Program
Volatile Organic Compounds Analysis

Volatile Organic Compounds	Percent Recovery*		Percent RPD*	
	Water	Soil	Water	Soil
MS/MSD				
1,1-Dichloroethene	61 to 145	59 to 172	14	22
Toluene	76 to 125	59 to 139	13	21
Trichloroethene	71 to 120	62 to 137	14	24
Benzene	76 to 127	66 to 142	11	21
Chlorobenzene	75 to 130	60 to 133	13	21
LCS				
Vinyl chloride	60 to 140	NA	---	---
1,2-Dichloroethane	60 to 140	NA	---	---
Carbon tetrachloride	60 to 140	NA	---	---
1,2-Dichloropropane	60 to 140	NA	---	---
Trichloroethene	60 to 140	NA	---	---
1,1,2-Trichloroethane	60 to 140	NA	---	---
Benzene	60 to 140	NA	---	---
cis-1,3-Dichloropropene	60 to 140	NA	---	---
Bromoform	60 to 140	NA	---	---
Tetrachloroethene	60 to 140	NA	---	---
1,2-Dibromoethane	60 to 140	NA	---	---
1,4-Dichlorobenzene	60 to 140	NA	---	---
Surrogate Compounds				
4-Bromofluorobenzene	86 to 115	59 to 113	---	---
4-Bromofluorobenzene [#]	80 to 120	---	---	---
1,2-Dichloroethane-d ₄	76 to 114	70 to 121	---	---
Toluene-d ₈	88 to 110	84 to 138	---	---

--- Not applicable
LCS Laboratory control sample
MS/MSD Matrix spike/matrix spike duplicate
NA Information not available

* The sources of this information are the USEPA CLP SOW (OLC01.0) for Low Concentration Water for Organics Analysis and the USEPA CLP SOW (OLM03.1) for Multi-media, Multi-concentration Organics Analysis. MS/MSD and surrogate criteria for the low concentration water method are not available; consequently, the limits shown were specified by Pace, Inc.

The listed limits are associated with the Low Concentration Water SOW.

**Table C2: Quality Control Performance Criteria for Matrix Spikes/Matrix Spike Duplicates, Laboratory Control Samples and Surrogates
U.S. Environmental Protection Agency Contract Laboratory Program
Semivolatile Organic Compounds Analysis**

Semivolatile Organic Compounds	Percent Recovery*		Percent RPD*	
	Water	Soil	Water	Soil
MS/MSD				
Phenol	---	26 to 90	---	35
2-Chlorophenol	---	25 to 102	---	50
1,4-Dichlorobenzene	---	28 to 104	---	27
N-Nitroso-di-n-propylamine	---	41 to 126	---	38
1,2,4-Trichlorobenzene	---	38 to 107	---	23
4-Chloro-3-methylphenol	---	26 to 103	---	33
Acenaphthene	---	31 to 137	---	19
4-Nitrophenol	---	11 to 114	---	50
2,4-Dinitrotoluene	---	28 to 89	---	47
Pentachlorophenol	---	17 to 109	---	47
Pyrene	---	35 to 142	---	36
LCS				
Phenol	44 to 120	NA	---	---
2-Chlorophenol	58 to 110	NA	---	---
4-Chloroaniline	35 to 98	NA	---	---
2,4,6-Trichlorophenol	65 to 110	NA	---	---
bis(2-Chloroethyl)-ether	64 to 110	NA	---	---
N-Nitroso-di-n-propylamine	34 to 102	NA	---	---
Hexachloroethane	32 to 77	NA	---	---
Isophorone	49 to 110	NA	---	---
1,2,4-Trichlorobenzene	44 to 96	NA	---	---
Naphthalene	56 to 160	NA	---	---
2,4-Dinitrotoluene	61 to 140	NA	---	---
Diethylphthalate	76 to 104	NA	---	---
N-Nitrosodiphenylamine	35 to 120	NA	---	---
Hexachlorobenzene	30 to 95	NA	---	---
Benzo(a)pyrene	55 to 92	NA	---	---
Surrogate Compounds				
Nitrobenzene-d ₅	35 to 114	23 to 120	---	---
2-Fluorobiphenyl	43 to 116	30 to 115	---	---
p-Terphenyl-d ₁₄	33 to 141	18 to 137	---	---
Phenol-d ₅	10 to 110	24 to 113	---	---
2-Fluorophenol	21 to 110	25 to 121	---	---
2,4,6-Tribromophenol	10 to 123	19 to 122	---	---
2-Chlorophenol-d ₄	33 to 110	20 to 130	---	---
1,2-Dichlorobenzene-d ₄	16 to 110	20 to 130	---	---
Nitrobenzene-d ₅ [#]	40 to 112	---	---	---
2-Fluorobiphenyl [#]	42 to 110	---	---	---

Table C2 (continued)

Semivolatile Organic Compounds	Percent Recovery*		Percent RPD*	
	Water	Soil	Water	Soil
P-Terphenyl-d ₁₄ #	24 to 140	---	---	---
Phenol-d ₅ #	17 to 113	---	---	---
2-Fluorophenol#	16 to 108	---	---	---
2,4,6-Tribromophenol#	18 to 126	---	---	---

--- Not applicable
 LCS Laboratory control sample
 MS/MSD Matrix spike/matrix spike duplicate
 NA Information not available

* The sources of this information are the USEPA CLP SOW (OLC01.0) for Low Concentration Water for Organics Analysis and the USEPA CLP SOW (OLM03.1) for Multi-media, Multi-concentration Organics Analysis.

Table C3: Quality Control Performance Criteria for Matrix Spikes/Matrix Spike Duplicates, Laboratory Control Samples, and Surrogates
U.S. Environmental Protection Agency Contract Laboratory Program
Chlorinated Pesticides and Polychlorinated Biphenyls Analysis

Pesticides and PCBs	Percent Recovery*		Percent RPD*	
	Water	Soil	Water	Soil
MS/MSD				
gamma-BHC (Lindane)	56 to 123	46 to 127	15	50
Heptachlor	40 to 131	35 to 130	20	31
Aldrin	40 to 120	34 to 132	22	43
Dieldrin	52 to 126	31 to 134	18	38
Endrin	56 to 121	42 to 139	21	45
4,4'-DDT	38 to 127	23 to 134	27	50
LCS				
gamma-BHC (Lindane)	56 to 123	NA	---	---
Heptachlor epoxide	74 to 150	NA	---	---
Dieldrin	33 to 130	NA	---	---
4,4'-DDE	50 to 150	NA	---	---
Endrin	56 to 121	NA	---	---
Endosulfan sulfate	50 to 100	NA	---	---
gamma-Chlordane	33 to 130	NA	---	---
Surrogate Compounds				
Tetrachloro-m-xylene	60 to 150	60 to 150	---	---
Decachlorobiphenyl	60 to 150	60 to 150	---	---

--- Not applicable
 LCS Laboratory control sample
 MS/MSD Matrix spike/matrix spike duplicate
 NA Information not available
 PCB Polychlorinated biphenyl
 RPD Relative percent difference

* The sources of this information are the USEPA CLP SOW (OLC01.0) for Low Concentration Water for Organics Analysis and the USEPA CLP SOW (OLM03.1) for Multi-media, Multi-concentration Organics Analysis.

Table C4: Quality Control Performance Criteria for Matrix Spikes/Matrix Spike Duplicates and Surrogates for Chlorinated Herbicides Analysis

Herbicides	Percent Recovery*		Percent RPD*	
	Water	Soil	Water	Soil
MS/MSD				
2,4-D	9 to 119	35 to 131	55	48
2,4-DB	84 to 102	84 to 102	30	50
Dicamba	21 to 115	57 to 121	47	32
Dichlorprop	91 to 103	91 to 103	30	50
2,4,5-T	67 to 103	67 to 103	30	50
2,4,5-TP	33 to 135	61 to 143	51	41
Surrogate Compounds				
DCAA	TBD	TBD	---	---

MS/MSD Matrix spike/matrix spike duplicate
 RPD Relative percent difference
 TBD To be determined

* Source: Environmental Science and Engineering, Inc. Comprehensive Quality Assurance Plan.

**Table C5: Quality Control Matrix Spike Matrix Spike Duplicates
Internal Standards and Recovery Standards,
U.S. Environmental Protection Agency SW-846 Method 8290
Polychlorinated Dibenzo-p-dioxin and Polychlorinated Dibenzofuran Analyses**

Dioxins and Furans	Mean Percent Recovery	
	Water	Soil
MS/MSD		
1,2,3,4,6,7,8-HpCDD	60 to 140	60 to 140
1,2,3,4,6,7,8-HpCDF	60 to 140	60 to 140
1,2,3,4,7,8-HxCDD	60 to 140	60 to 140
1,2,3,4,7,8-HxCDF	60 to 140	60 to 140
1,2,3,7,8-PeCDD	60 to 140	60 to 140
2,3,4,7,8-PeCDF	60 to 140	60 to 140
2,3,7,8-TCDD	60 to 140	60 to 140
2,3,7,8-TCDF	60 to 140	60 to 140
OCDF	60 to 140	60 to 140
OCDD	60 to 140	60 to 140
Internal Standards		
¹³ C ₁₂ -2,3,7,8-TCDD	40 to 135	40 to 135
¹³ C ₁₂ -2,3,7,8-TCDF	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,7,8-PeCDD	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,7,8-PeCDF	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	40 to 135	40 to 135
¹³ C ₁₂ -OCDD	40 to 135	40 to 135
Recovery Standards		
¹³ C ₁₂ -1,2,3,4-TCDD ^a	40 to 135	40 to 135
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD ^b	40 to 135	40 to 135

- a. Used for recovery determinations of TCDD, TCDF, PeCDD, and PeCDF internal standards.
b. Used for recovery determinations of HxCDD, HxCDF, HpCDD, HpCDF, and OCDD internal standards.

Table C6: Quality Control Performance Criteria for Matrix Spikes and Laboratory Duplicates and Laboratory Control Samples^a
U.S. Environmental Protection Agency
Contract Laboratory Program Inorganics Analysis

Analyte	Matrix Spike and Laboratory Duplicate			
	% Recovery ^c		% RPD ^c	
	Water	Soil	Water ^d	Soil ^e
Aluminum	75 to 125	--- ^b	± 20	± 20
Antimony	75 to 125	75 to 125	± 20	± 20
Arsenic	75 to 125	75 to 125	± 20	± 20
Barium	75 to 125	75 to 125	± 20	± 20
Beryllium	75 to 125	75 to 125	± 20	± 20
Cadmium	75 to 125	75 to 125	± 20	± 20
Calcium	--- ^b	--- ^b	± 20	± 20
Chromium	75 to 125	75 to 125	± 20	± 20
Cobalt	75 to 125	75 to 125	± 20	± 20
Copper	75 to 125	75 to 125	± 20	± 20
Cyanide	75 to 125	75 to 125	± 20	± 20
Iron	75 to 125	--- ^b	± 20	± 20
Lead	75 to 125	75 to 125	± 20	± 20
Magnesium	--- ^b	--- ^b	± 20	± 20
Manganese	75 to 125	75 to 125	± 20	± 20
Mercury	75 to 125	75 to 125	± 20	± 20
Nickel	75 to 125	75 to 125	± 20	± 20
Potassium	--- ^b	--- ^b	± 20	± 20
Selenium	75 to 125	75 to 125	± 20	± 20
Silver	75 to 125	75 to 125	± 20	± 20
Sodium	--- ^b	--- ^b	± 20	± 20
Thallium	75 to 125	75 to 125	± 20	± 20
Vanadium	75 to 125	75 to 125	± 20	± 20
Zinc	75 to 125	75 to 125	± 20	± 20

--- Not applicable

RPD Relative percent difference

- Laboratory control sample recoveries for water and soil shall meet the control limits established for the particular LCS batch analyzed. For water analyses, a control limit of ± 20 percent of the true value must be used if no control limits are provided with the LCS solution.
- No spike required when analyzed by ICP methods, otherwise limits of 75 to 125 apply.
- The sources of this information are the USEPA CLP SOW (ILC01.0) for Low Concentration Water for Inorganics Analysis and the USEPA CLP SOW (ILM03.0) for Multi-media, Multi-concentration Inorganics Analysis.
- A control limit of ± the Instrument Detection Limit (IDL) is used if either the sample or the duplicate value is less than 5 x IDL.
- A control limit of ± the Contract Required Detection Limit (CRDL) is used if either the sample or duplicate value is less than 5 x CRDL.

Table C7: Quality Control Performance Criteria for Matrix Spikes and Laboratory Duplicates for Total Organic Carbon and Total Petroleum Hydrocarbons Analysis

Analyte	Matrix Spike and Laboratory Duplicates			
	Percent Recovery ^a		Percent RPD ^a	
	Water	Soil	Water	Soil
Total organic carbon (415.1 ^b /9060 ^c)	87 to 113	82 to 116	13	17
Total Petroleum Hydrocarbons (Modified 8015)				
Gasoline	TBD	TBD	TBD	TBD
Diesel	32 to 120	58 to 121	40	20
Motor oil	TBD	TBD	TBD	TBD

--- Not applicable
TBD To be determined

- These ranges are advisory ranges only. Failure to achieve these recovery values will not initiate reanalysis.
- U.S. Environmental Protection Agency. 1983. Methods for Chemical Analysis of Water and Wastes. March.
- U.S. Environmental Protection Agency. 1994. Test Methods for Evaluating Solid Waste - Physical/Chemical Methods SW-846.

**Table C8: Quality Control Goals
Polychlorinated Biphenyl Field Screening**

PCB Field Screening		Goal
Duplicate Sample Agreement		Agreement between duplicate samples regarding presence or absence of PCBs RPD ≤ 30
Confirmatory Laboratory Analysis		Agreement between PCB soil screening result and laboratory analysis regarding presence or absence of PCBs RPD ≤ 30
Matrix Spike Samples		Confirmation of positive soil screening result for soil samples with added PCBs
<hr/>		
PCB	Polychlorinated biphenyls	
RPD	Relative Percent Difference	

Appendix D
RESPONSES TO COMMENTS

**U.S. ENVIRONMENTAL PROTECTION AGENCY (RCRA) COMMENTS (DATED MAY 11, 1995)
REGARDING THE DRAFT PHASE II RCRA FACILITY INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN FOR FORT BENJAMIN HARRISON,
MARION COUNTY, INDIANA (IN4 210 090 003) - JUNE 1995**

GENERAL COMMENTS

Comment

In discussions between the Army, the State of Indiana, and the U.S. EPA, the problems associated with having separate QAPPs for the base closure program and the RCRA corrective action program were identified and discussed. The problems stem from the fact that, while the RFI is being done as a requirement under the RCRA permit, the data also needs to be acceptable under CERCLA requirements in order that the property can be transferred to non-federal authorities. In a worst case situation, the data would be accepted under the RCRA program, but found to be unacceptable under CERCLA guidance, and thus require additional sampling and analysis before the facility could be transferred. This would result in the expenditure of significant additional time and resources. Even trying to adapt the RCRA QAPP to meet the CERCLA requirements would likely be costly in terms of both time and resources.

To alleviate the QAPP problems, the RCRA program is willing to accept a CERCLA approved QAPP, reserving the right to comment on the QAPP and requiring the Army to respond to any identified concerns. We believe that the CERCLA QAPP can adequately address RCRA corrective action requirements as long as the data quality objectives are equivalent, the list of constituents include all RFI constituents of concern, and the detection limits of the analytical procedures are sufficient to allow the data to be used in preparing an acceptable risk assessment. Both the Army, the BRAC team contact, and the State of Indiana indicated that having a single QAPP would be greatly preferable to having 2 separate QAPPs for the RCRA SWMUs. Therefore, we have decided not to review the RCRA QAPP submitted as part of the draft workplan. We will review the CERCLA QAPP, when it is submitted. If it meets RCRA data quality objectives it will be approved for use in the Phase II RFI.

Response

Comment noted.

**U.S. ENVIRONMENTAL PROTECTION AGENCY (CERCLA) COMMENTS
(DATED JUNE 5, 1995) REGARDING THE DRAFT QUALITY ASSURANCE PROJECT PLAN
FOR PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
JUNE 1995**

GENERAL COMMENTS

Comment No. 1

Is the current document in EPA format? We concur.

Response

The Quality Assurance Project Plan (QAPP) referenced in these comments was a revision of the Phase I Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) QAPP prepared following U.S. Environmental Protection Agency (EPA) Region V RCRA guidance for the preparation of QAPPs. After the draft QAPP was submitted, personnel from the EPA Region V RCRA and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) programs decided that the Phase II RFI analytical program would comply with CERCLA requirements. Therefore, the draft Phase II RFI QAPP was revised to address EPA Region V CERCLA guidance for the preparation of QAPPs. A second draft Phase II RFI QAPP was submitted to the regulatory agencies on June 16, 1995. The responses below are based on the second draft Phase II RFI QAPP.

Comment noted.

Comment No. 2

Section 9.2.2.1, how was independent data validation done? This section states that the independent data validation was done consistently with the U.S. EPA Functional Guidelines for Inorganics Analysis (1988), and Organics Analysis (1991c). However, Data Flagging codes are not consistent with U.S. EPA Functional Guidelines. Also, there were no changes to the Environmental Investigation data validation (United States Army Environmental Center [USAEC] SOP-Chem-012).

Response

The Phase II analytical results will be validated following the procedures provided in the respective 1994 releases of the EPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review and the EPA CLP National Functional Guidelines for Inorganic Data Review. Data qualifiers applied to the Phase II analytical results will be consistent with the qualifiers in the EPA Functional Guidelines.

Comment No. 3

Table 1.2 Data Quality Objectives (DQOs)? The Table must be re-written in order to specify the DQOs for each analyses.

Response

The QAPP has been revised to include an expanded discussion of data quality objectives (DQOs). An additional table (Table 1.4) has been included to specify DQOs for each analysis.

Comment No. 4

Does standard operating procedures (SOPs) outline U.S. EPA's standard limits? Does the QAPP specify SW-846 calibration methods and standards? This issue has not been addressed. The full list of analytical laboratory SOPs are not provided with the Revised April 1995 QAPP. Also, in Section 11.13, the calibration of field instruments are not addressed to U.S. EPA standards. In Section 6.2.1, USAEC Class 1, 1P, and 1M analytical methods and calibration methods were to be used instead of SW-846. No specific calibration SOPs were set forth; the text refers to the USAEC Chemistry Branch for guidance.

In Section 6.2.1.2, the calibration standards do not meet SW-846 standards, nor do they use the System Performance Check Compound (SPCC) or Calibration Check Compound (CCC) standards. In Section 6.2.1.3, SW-846 is cited for continuous calibration (e.g., if calibration fails, the testing stops immediately and repeat calibration. If there is secondary failure, would implement corrective actions, and retest all samples from last valid calibrations point on), but, calibration standards stated in Section 6.2.1.2 are not valid.

Response

The final QAPP specifies CLP analytical methods except for herbicides, dioxin/furans, total organic carbon, total petroleum hydrocarbons, and cation exchange capacity, which are SW-846 methods. CLP methods will follow Standard Operating Procedures (SOPs) specified by the EPA's CLP. An analytical-specific SOP for each SW-846 method used, which includes specifications for instrument calibration and standards, is included in the final QAPP.

Comment No. 5

MS/MSD Matrix Spikes - Are they field material or lab generated? What about surrogates? This issue has been partially addressed. What are "natural" versus standard surrogate spikes? What are specific URLs for particular compounds? Table 8.2 needs clarification, and comparison to U.S. EPA not USAEC classes and analytical methods. SW-846 surrogates are mentioned; which ones for what method? What are the surrogate recovery rates?

Response

Matrix spike/matrix spike duplicate (MS/MSD) samples collected in the field will be analyzed during the Fort Benjamin Harrison (FBH) Phase II investigations. These MS/MSD samples will be collected at a frequency of 5 percent of the number of investigative samples collected.

Quality control (QC) criteria, including MS/MSD recoveries, surrogate recoveries, and frequency of laboratory QC samples, will be consistent with the QC criteria of each respective CLP or SW-846 method followed by the laboratory.

Comment No. 6

Appendix E, Table E-2? The Reporting Limits do not match SW-846 Method 8540B, 8270B, (8080, 8150B, 8290B EQLs approximate), nor the U.S. EPA Region 5 Model QAPP Quantitation Limits. The referenced source of Reporting Limits is from Environmental Science and Engineering, Inc. Did they get the reporting limits from older versions of SW-846 methods?

Response

Reporting limits, which were provided in the QAPP reviewed for this comment, are laboratory-determined reporting limits. The reporting limits were determined as specified in Appendix B, Part 136, of Volume 40 of the Code of Federal Regulations. Method detection limits will be validated for each method before Phase II RFI Samples are analyzed. Method detection limits will be assessed following the procedures described in 40 Code of Federal Regulations (CFR) Part 136, Appendix B "Definition and Procedure for the Determination of the Method Detection Limit" The following text has been added to Section 7.0 of the QAPP:

"Before an analytical method can be used for this project, the subcontractor laboratory must demonstrate the ability to perform the method for the specified analytes. The laboratory will determine an MDL for all analytes of interest using the procedures described in 40 CFR, Part 136, Appendix B (EPA 1984). These MDL procedures are summarized as follows:

- The laboratory will prepare a standard matrix sample at one to five times the estimated MDL.
- Seven aliquots of the sample will be processed through the entire method.
- The laboratory will calculate the standard deviation of results from the seven replicate samples.
- The MDL is equal to the standard deviation multiplied by the Student's t value (3.143).

The MDL will be equal to or less than the respective EPA estimated quantitation limits (EQLs) or contract required detection limits (CRDLs) for EPA-SW-846 and CLP methods respectively."

**U.S. ARMY ENVIRONMENTAL CENTER CHEMISTRY BRANCH
COMMENTS (DATED JUNE 10, 1995) REGARDING
THE DRAFT QUALITY ASSURANCE PROJECT PLAN FOR
PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
JUNE 1995**

GENERAL COMMENT

Comment

In general, data validation is the responsibility of the prime contractor. although specific comments are listed below, the contractor should review the entire document and remove statements that say that USAEC reviews NTAMs data, requires control charts, method performance documentation, etc.

Response

The Quality Assurance Project Plan for the Phase II RCRA Facility Investigation, Fort Benjamin Harrison, Marion County, Indiana (QAPP) has been revised. Text indicating that the U.S. Army Environmental Center (USAEC) reviews Non-Thama Approved Methods (NTAMs) data, requires control charts, method performance documentation, and related statements have been removed from the QAPP.

SPECIFIC COMMENTS

Comment No. 1, Section 3.2, second paragraph

The accuracy, precision, sensitivity requirements, and detection limits should be found in Appendix A. The procedure for validation the detection limits should also be included. Provide information.

Response

A summary of the quality control criteria, including those for accuracy, precision, sensitivity, and reporting limits, for the respective analytical methods are provided in Appendix C of the QAPP. Quality control criteria for EPA SW-846 analyses including herbicides, dioxins/furans, total petroleum hydrocarbons, and total organic carbon will be included in the laboratory's Standard Operating Procedures included in Appendix A. Quality control criteria for routine analytical services (RAS) parameters including volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), pesticides/polychlorinated biphenyls (PCBs), and metals and cyanide are specified in the current statement of work (SOW). Accuracy and precision requirements for field screening analyses are also included in Appendix C of the QAPP.

Method detection limits will be validated for each method before Phase II RFI Samples are analyzed. Method detection limits will be assessed following the procedures described in 40 Code of Federal Regulations (CFR) Part 136, Appendix B "Definition and Procedure for the Determination of the Method Detection Limit" The following text has been added to Section 7.0 of the QAPP:

"Before an analytical method can be used for this project, the subcontractor laboratory must demonstrate the ability to perform the method for the specified analytes. The laboratory will determine an MDL for all analytes of interest using the procedures described in 40 CFR, Part 136, Appendix B (EPA 1984). These MDL procedures are summarized as follows:

- The laboratory will prepare a standard matrix sample at one to five times the estimated MDL.
- Seven aliquots of the sample will be processed through the entire method.
- The laboratory will calculate the standard deviation of results from the seven aliquots.
- The MDL is equal to the standard deviation multiplied by the Student's t value (3.143).

The MDL will be equal to or less than the respective EPA estimated quantitation limits (EQLs) or contract required detection limits (CRDLs) for EPA-SW-846 and CLP methods respectively."

Comment No. 2, Section 5.2.1

Lot sizes for NTAMS data do not need to be based on the rate limiting step of a method. This Center recommends daily lots. The definition of the lot size should be worked out with the subcontractor laboratory. Clarify lot size.

Response

Lot sizes for NTAMS data will not be based on the rate limiting step of the method. The criteria for lot size described in Section 5.2.1 of the draft QAPP have been revised.

Comment No. 3, Section 5.3

Shouldn't the "evidence file" be forwarded to AEC when the project is complete?

Response

Section 5.3 has been revised to indicate that the USAEC is the custodian of the all documents and information related to the FBH Phase II RFI.

Comment No. 4, Section 6.3.3

Remove references to results of the SARMS characterization must be sent to USAEC with method documentation package.

Response

The following sentence has been deleted from Section 6.3.3 the QAPP:

"The characterization analyses must be performed before method validation is initiated, and the results must be provided to USAEC with the Method Documentation Package."

Comment No. 5, Section 8.3.2

Clarify what the QC check sample is, where it comes from, and what the standard matrix is.

Response

Text in Section 8.2.2 (Formerly Section 8.3.2) does not specifically pertain to an individual quality control sample, but rather pertains to method specified internal laboratory quality control samples including calibration standards, calibration verification check standards, method blanks, blank spiked samples, laboratory duplicates or replicates, surrogate spikes and matrix spiked samples. The internal laboratory quality control samples are distinct from external quality control samples. Internal quality control samples are specified in the CLP SOW and in the SOP for herbicides (Appendix A). The text in this section has been revised as follows to clarify the role of internal laboratory quality control samples.

Section 9.2.2.1

- a. 1st para. USAEC will not perform data review on NTAMs data. Revise.
- b. 1st bullet. USAEC will not routinely look at Fort Ben Harrison data on lab audits. If problems arise, they will be discussed but NTAMs data will not automatically be reviewed.
- c. 2nd bullet. The only time USAEC will routinely look at QC results is if an error is generated. Since NTAMs data are not checked for such criteria, AEC will not be looking at such QC samples. Revise.
- d. 3rd bullet states that 100% of data will be validated. The following para. states that 20% of sample results will be checked. This appears to be contradictory. AEC recommends that you propose the minimum amount of validation that the state of Indiana and Region V may accept. This would be the start of negotiations with AEC and the regulators. Specify as little validation as possible. Additionally, specify the percentages of validation of each item, ie. surrogate recoveries, GC/MS tuning, lab control sample results, etc.

Response

- a. The second sentence in the first paragraph of Subsection 9.2.2.1 has been revised to remove reference to USAEC.
- b. The first bullet of Section 9.2.2.1 has been revised.
- c. The second bullet of Section 9.2.2.1 has been revised.
- d. The paragraph following the third bullet of Section 9.2.2.1 describing data validation has been revised as follows to more clearly indicate the data validation level of effort.

Comment No. 6, Section 9.4.2

Method accuracy will always be 1 therefore there will be no correction. Remove "method accuracy" in the 3rd sentence, 1st para.

Response

The third sentence, first paragraph of Section 9.4.2 has been revised as requested

Comment No. 7, Section 9.4.2.1

This section needs to be revised. NTAMS data are not electronically checked as rigorously as the approved methods are.

- *Electronic validation (chemistry related issues) for NTAMS data includes the following:*
 - a. *test name, lab code, installation code, prime contractor code are valid*
 - b. *test name valid for method*
 - c. *units match matrix*
 - d. *holding times are met*
 - e. *dates checked, ie. analysis date after extraction date, sampling date, etc.*
- *NTAMS data are not check for*
 - a. *if all test names in a method were analyzed for.*
 - b. *no QC checking, ie MS/MSD, method blanks, etc.*
 - c. *Any value be entered - no check for MDL or upper reporting limit.*

Response

Section 9.4.2.1 has been revised as follows to restrict the discussion of the electronic NTAMS data review.

The IRDMIS group check assesses whether all station identifications for the lot data exist in the map file for the appropriate installation."

Comment No. 8, Section 9.5 and 9.6

This Center recommends CLP data packages therefore these sections need to be modified.

Response

Section 9.4.2.2 (Hardcopy Data Deliverables) has been revised to indicate that hardcopy and electronic versions of CLP data packages will be submitted to HLA and USAEC.

Section 9.5 has been deleted.

Section 9.6 (New Section 9.5) has been revised to restrict discussion to elements of the CLP data package format.

Comment No. 9, Section 10.1.1.2

Qualify the field audit requirement by AEC or designated representative with the term "if resources permit".

Response

The first sentences of Section 10.1.1.1 and Section 10.1.1.2 have been modified as requested.

Comment No. 10, Section 10.2.1.3

The army looks at data generated from approved methods and will not be looking at NTAMs data. The laboratory may contact the AEC if problems arise but NTAMs packages will not be routinely reviewed on lab audits. The section concerning AEC audits should be deleted.

Response

The subsection "U.S. Army Environmental Center Laboratory Audits" under Section 10.2.1.3 has been deleted.

Comment No. 11, Section 12.1

The word "assess" should be "assessed".

Response

The word assess, in the next to the last sentence of the first full paragraph of Section 12.1, has been revised to assessed.

Comment No. 12, Section 14.1.1.

"QC" charts are not required to be submitted to this Center. However, if the laboratory routinely generates them for internal purposes, they should continue to generate them.

Response

Section 14.1.1 has been revised by deleting reference to submittal of control charts to USAEC.

Comment No. 13, Section 14.2

Remove this section.

Response

Section 14.2, Frequency of Quality Assurance Reports" has been revised by removing the requirement for QA report submittal to USAEC.

Comment No. 14, Section 14.3

Remove all references to control charts.

Response

Reference to laboratory QC charts has been removed from Section 14.3 of the QAPP.

**IDEM COMMENTS (DATED JUNE 23, 1995) REGARDING THE
DRAFT QUALITY ASSURANCE PROJECT PLAN
FOR THE PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
APRIL 1995**

GENERAL COMMENTS

Comment No. 2

The Army is currently revising the April 1995 RFI Phase II QAPP. The QAPP will be based on the Region V CERCLA Model QAPP. IDEM did not review the April 1995 QAPP, but we will review the revised QAPP.

Response

Comment noted.

Comment No. 3

IDEM Defense Environmental Restoration Program and the EPA Federal Facilities Branch raised concerns regarding the use of the Quality Assurance Project Plan for FBH RCRA Facility Investigation, Vol. I & II, December 1993, for the work conducted on the Environmental Investigation sites at FBH. The December 1993 QAPP was a hybrid QAPP using a combination of RCRA, CERCLA and Army methods. This has caused a great of confusion during the Phase I EI. Karen Mason-Smith, EPA Federal Facilities Branch, and myself have requested that the Army use the CERCLA Region V model QAPP to develop the Phase II EI QAPP for all EI activities.

Response

A separate Quality Assurance Project Plan (QAPP) based on the U.S. Environmental Protection Agency (EPA) Region V Model Superfund QAPP was prepared for the Phase II Environmental Investigation (EI) activities and was submitted to IDEM on November 17, 1995.

Comment No. 4

The RCRA Facility Investigation (RFI) is being conducted under the RCRA rules and regulations and the Environmental Investigation (EI) is being conducted under the CERCLA rules and regulations. The April 1995 QAPP was prepared for Mr. Gale Hruska, EPA RCRA Corrective Action, for use on the Phase II RFI. Even though the RFI must be conducted under the RCRA rules and regulations, all actions taken must satisfy base closure property transfer requirements under CERCLA. In an effort to satisfy the CERCLA and RCRA requirements and alleviate future problems, the RCRA program agreed to accept a CERCLA approved QAPP, reserving the right to comment on the QAPP and requiring the Army to respond to any identified concerns for the RCRA program.

Response

The U.S. Department of the Army (Army) submitted a revised Draft Phase II Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) QAPP to the Indiana Department of Environmental Management (IDEM) and EPA Region V on June 16, 1995. This QAPP was based on the EPA Region V Model Superfund QAPP.

Comment No. 5

Mr. Gale Hruska, the EPA RCRA Corrective Action Project Manager, gave Bill Nelson, AEC, verbal acceptance on May 1, 1995, for the Army to utilize a CERCLA Quality Assurance Project Plan (QAPP) for the Phase II RFI at FBH. The Base Realignment and Closure Cleanup Team felt that using the CERCLA QAPP could fulfill both RCRA and CERCLA requirements as long as the EI and RFI data quality objectives were clearly stated and the appropriate detection limits were used and reported.

Response

As indicated in the response to Comment 4, the Army submitted a revised Draft Phase II RFI QAPP to IDEM and EPA Region V for review and comment. The QAPP identifies data quality objectives (DQOs) and analytical detection limits. Analytical methods specified in the revised QAPP include EPA Contract Laboratory Program (CLP) and SW-846 methods. The respective CLP contract required quantitation limits and SW-846 estimated quantitation limits are included in the QAPP.

Comment No. 6

The Base Realignment and Closure Cleanup Team Karen Mason-Smith, Gale Hruska, Richard Blume-Weaver, Bill Nelson, and myself agreed on May 1, 1995, to use the CERCLA Region V Model QAPP as a model to develop the FBH Phase II RFI and Phase II EI QAPPs. This decision rendered the April 1995 QAPP, that had been prepared for the Phase II RFI, unusable. Therefore IDEM did not review the Quality Assurance Project Plan (QAPP) for FBH, distributed in April 1995. The Army agreed to produce two QAPP's, one for the RFI Phase II and one for the EI Phase II, using the EPA Region V CERCLA Model QAPP as the model. Data Quality objectives should be specified to met programmatic requirements. Both EPA RCRA, EPA CERCLA and IDEM will work together to assure the requirements under both RCRA and CERCLA are met. The sampling analyte list should include all RCRA and CERCLA parameters of concern. A table should be developed listing the constituents, the analytical method to be used, the detection limit, and the MCL or action level if known.

Response

Separate QAPPs for the respective Phase II RFI and EI field investigations were prepared. Both QAPPs are based on the EPA Region V Superfund Model QAPP.

A draft version of the Phase II RFI QAPP was submitted to IDEM and EPA on June 16, 1995, for review and comment. This QAPP includes a summary of Phase II RFI DQOs, based on the "boiler-plate" language specified in Section 1.6 of the EPA Region V Superfund Model QAPP. (The "boiler-plate" language has been pre-approved by the EPA Region V Quality Assurance Section.) However, on the basis of specific IDEM comments regarding DQOs, the DQO summary will be expanded to include specific analytes, media, and associated DQOs.

The Draft Phase II RFI QAPP includes analytical methods and RCRA parameters of concern. The analytical methods and RCRA parameters of concern were defined and agreed upon by EPA Region V and the Army during a March 30, 1995, and a December 12, 1995 meeting in Chicago, Illinois.

Table 1.4 has been added to Section 1 of the Phase II RFI QAPP. This table provides a summary, by site and by medium; of the proposed Phase II RFI sampling program, and the respective analyses, DQOs, and risk-based action levels.

**U.S. ARMY CENTER FOR HEALTH PROMOTION AND PREVENTIVE
MEDICINE COMMENTS (DATED JULY 24, 1995) REGARDING
THE DRAFT QUALITY ASSURANCE PROJECT PLAN FOR
PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
JUNE 1995**

SPECIFIC COMMENTS

Comment No. 1, page 5-4, Section 5.1.3, paragraph 1, Mr. McKenzie Field Logbooks and Documentation

If an incorrect entry is made, the information will be crossed out with a single strike mark. The entry should also be initialed and dated. A comment as to why the entry was crossed out should also be made.

Recommendation: Change the paragraph to reflect the comment.

Response

The third paragraph of Section 5.1.3 has been modified to reflect the comment.

Comment No. 2, page 9-2, Section 9.1.2, paragraph 2, Mr. McKenzie Laboratory Data Reduction Procedures

Corrections should be made by drawing one line through the incorrect entry, entering the correct information, initialing, and dating the change. A comment as to why the entry was crossed out should also be made.

Recommendation: Change the paragraph to reflect the comment.

Response

The third full paragraph of Section 9.1.2 has been modified to reflect the comment.

**IDEM COMMENTS (DATED OCTOBER 24, 1995) REGARDING
THE DRAFT QUALITY ASSURANCE PROJECT PLAN FOR
PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
JUNE 1995**

GENERAL COMMENT

Comment No. 1, Signature page

IDEM Add the following names:

Richard Blume-Weaver, Base Environmental Coordinator

Manuela Johnson, Indiana Department of Environmental Management Quality Assurance/Quality Control Officer, Chemistry Section Chief

Karen Mason-Smith, U.S. Environmental Protection Agency Project Coordinator

Response

The signature page has been revised to include the requested additional signatories.

Comment No. 2, Page 1-3, 1st full paragraph, last sentence

RFI Environmental concerns at the former sanitary landfill (west side of the base) were addressed by the U.S. Army Corps of Engineers (COE) under the IDEM Solid Waste Program.

IDEM The sentence should read as follows: "Environmental concerns at the former sanitary landfill (west side of the base) are currently being addressed by the U.S. Army Corps of Engineers (COE) under the IDEM Solid Waste Program."

Response

The sentence has been revised as requested.

Comment No. 3, Page 1-5, Section 1.2

IDEM Include the following as objectives:

Site characterization;

Determine nature and extent of contamination for property transfer;

Provide enough data to conduct a human health and ecological risk assessment;

Provide enough data to evaluate remedial alternatives.

Response

IDEM requested that additional objectives be included in Section 1.2. These additional specific objectives, or their equivalents, are already listed in Section 1.2.

Comment No. 4, Page 3-3, Section 3.3

RFI It is expected that Environmental Science and Engineering, Inc. will provide data meeting QA acceptance criteria for 80 percent or more for all samples tested using the RAS and methods listed in Section 3.1 of this QAPjP.

IDEM Since the contractors are required to provide 100% data validation, the QA acceptance criteria will change. Please change the percentage to the appropriate number.

Response

Section 3.3 (page 3-4) has been revised to clarify data validation and data completeness. Section 3.3 has been revised to specify that 90 percent or more of the data generated by the laboratory is expected to meet the QC acceptance criteria specified in the QAPjP.

Comment No. 5, Page 6-9, Section 6.2.4

RFI Inorganics - The analytical method was referred to as CLP SOW OLMO3.1.

IDEM The analytical method should be CLP SOW ILMO3.0, please correct.

Response

The inorganic analytical method (Section 6.2.2.4, page 6-11) has been corrected to identify analytical Method CLP SOW ILM03.0 or ILC01.0, as appropriate for the sample medium.

Comment No. 6, Page 6-11, Section 6.3.1, last sentence

RFI The use of secondary standards is encouraged as a conservation method for the more costly standard analytical reference materials (SARM's).

IDEM If the secondary standards are used, the Army must offer validation that acceptable percent recoveries are met, especially for lower concentration values and lower concentration protocols.

Response

The laboratory will be permitted to use secondary standards that, as indicated in the QAPjP, are traceable to National Institute of Technology (NIST) standard analytical reference materials. The laboratory will maintain documentation regarding the purity of each secondary standard.

Comment No. 7, page 7-1, 1st paragraph

IDEM IDEM requires quantitation/detection limits (lower than those indicated in Tables 3.1 - 3.7) for water samples. Analytical methods should provide detection limits in water that are lower than the

State and Federal drinking water limits for compounds of interest. If the lower quantitation/detection limits require Special Analytical Services (SAS), it should be discussed in this Chapter.

Response

The analytical methods for water have been revised to the CLP SOW methods (OLC01.0) for low concentration water analyses, and Tables 3.1, 3.2, 3.3, and 3.5 have been updated with the lower CRQLs and CRDLs for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), pesticides, and polychlorinated biphenyls, and metals and cyanide.

Comment No. 8, Page 7-1, Section 7.1, 1st paragraph

RFI All samples for CLP TCL dioxins/furans will be analyzed according to analytical procedures set forth in the EPA CLP SOW DELMO1.0.

IDEM The dioxins/furans method on Page 7.1 refers to CLP SOW DELMO1.0 analytical method, yet Page 3-3, section 3.2 and Table 7.1 refers to the dioxins/furans method as CLP SOW DELMO1.1. Please correct the method number so that the method listed is correct and consistent.

Response

The Army, in response to a request by IDEM (3/22/96 Conference call to discuss IDEM 3/15/96 comments) will analyze samples for dioxins/furans using EPA's SW-846 8290.

Comment No. 9, Page 7-2, Section 7.2

IDEM This section should include the procedures for field measurements of pH, Eh, specific conductivity, temperature and any other parameter described in the Technical Sampling Plan.

Response

Section 7.2 has been expanded to include brief descriptions of procedures for field measurements proposed in the Phase II RFI field program including pH, Eh, specific conductance, and temperature.

Comment No. 10, Section 7.4.2, 1st sentence

RFI Soil samples consist of subsurface soil.

IDEM The sentence should state soil samples consist of surface, subsurface, and sediment soil.

Response

The description of soil samples has been revised (current Section 7.3.2) to indicate that soil samples include surface and subsurface soil and sediment.

Comment No. 11, Page 8-1, Section 8.1.1

IDEM Add the measuring parameters of cation exchange capacity, Eh and total organic carbon to this list.

Response

Redox potential (Eh) in groundwater has been added to the list of parameters to be measured in the field (Section 8.1.1). Cation exchange capacity (soil) and total organic carbon (soil and water) are not performed as field measurements. However, these parameters have been added to the list of analyses for soil and groundwater samples (Table 7.1).

Comment No. 12, Page 9-2, Section 9.1.2, 1st paragraph

RFI Samples collected at FBH for Level IV analyses will be sent to the subcontract laboratory.

IDEM The laboratory should be an approved CLP laboratory.

Response

IDEM was contacted regarding this comment. Ms. Lorraine Wright indicated that the laboratory selected for analysis of samples collected during the Phase II RFI field program does not need to be an active participant in the CLP program. However, the laboratory selected for analysis of the FBH Phase II RFI samples shall adhere to all CLP criteria and provide CLP-type deliverables for the analyses performed.

Comment No. 13, Page 9.2, Section 9.1.2, 1st paragraph

RFI Data reduction, evaluation, and reporting for samples analyzed by the analytical laboratory will be performed according to specifications outlined in the CLP RAS SOW (OLMO3.1) or the most current for the organics and SOW (ILMO3.0) or the most current version for inorganics.

IDEM IDEM requires that the Army use the Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for residential well water samples for the FBH water samples. The lower quantitation limits should allow for the detection of contaminants at or below the Maximum Contaminate Levels (MCLs).

Response

The analytical methods for water have been revised to require CLP SOW methods (OLC01.0 and ILC01.0) for low concentration water analyses, and the tables have been revised with the lower CRQLs and CRDLs from these methods. However, the MCLs for several compounds including benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, dibromochloropropene, indeno(12,3-cd)-pyrene and PCBs are lower than their respective CRQLs for the low concentration water analyses.

Comment No. 14, Page 9-5, 1st paragraph

RFI Additional specific evaluation of data critical to the integrity of the decision-making process for the RFI will be performed on 10 percent of the data.

IDEM IDEM's Office of Environmental Response Quality Assurance Officer is requiring the Army to conduct 100% data validation of all data. Please change the document to reflect that the Army will complete a 100% validation of all data.

Response

Text in Section 9.2.1 (first paragraph, page 9-3) has been revised to indicate that

"Verifications of field procedures will be performed on 100 percent of the following field data:"

Comment No. 15, Page 9-8, 1st paragraph

RFI Validation of the RFI analytical data will follow procedures consistent with EPA Functional Guidelines for Inorganics and Organic Analytes [EPA 540/R-94/012, February (EPA, 1994a,b)].

IDEM Add "or most current version."

Response

The text (page 9-8) has been revised as requested.

Comment No. 16, Page 9-8, 3rd bullet and 1st paragraph

RFI 3rd bullet: "Hardcopy data validation - HLA will validate 100 percent of the data produced for each method during the RFI analytical program."

1st paragraph: "Twenty percent of the sample results within each lot will be checked, and the results of the validation summarized."

IDEM Both of the statements above refer to data validation. One refers to 100 percent data validation of each method; the other refers to 20% of each lot will be checked and then the 20% validated. IDEM's Office of Environmental Response Chemistry Section is requiring the Army to conduct 100% data validation of all data. Please change the document to reflect that the Army will complete a 100% validation of all data.

Response

The paragraph following the third bullet of Section 9.2.2.1 describing data validation has been revised to more clearly indicate the data validation level of effort.

Comment No. 17, Page 9-10, Section 9.3

RFI Statistical methods will be used to evaluate concentrations of target analytes in background and investigative site samples collected at FBH.

IDEM IDEM recommends incorporating the new background/upgradient samples, collected during the Phase II Environmental Investigation, be incorporated in the RCRA Facility Investigation such the EI and the RFI will be evaluating the sites based on the same background information. The Phase I and Phase II background data sets should be evaluated to see if they are similar before combining data from the two sets. Groundwater background/upgradient samples from Phase I RFI and Phase II EI should not be combined.

Response

The Army submitted a description of the proposed Phase II background sampling program to IDEM and EPA on October 13, 1995. A revised version of this plan was discussed during an April 4, 1996, telephone conference and approved by IDEM and EPA. The final version of this plan is included in the Final TSP for the Phase II Environmental Investigation. The plan includes a general description of proposed surface and subsurface soil background sampling and upgradient groundwater sampling. Although intended to be part of the EI, the Phase II background sampling program will be implemented concurrently with the Phase II RFI field program.

The background sample collection plan indicates that the analytical results from those Phase I background soil samples previously accepted by IDEM will be combined with the Phase II background soil sample results to define background at FBH. The respective Phase I and Phase II background soil data will be evaluated (and agencies consulted) to assess whether the data are similar before the data sets are combined. Because of changes in the analytical methods required by IDEM and the EPA Region V CERCLA program, some differences in the Phase I and Phase II soil background results may occur. Because statistical analysis of groundwater will not be performed, the Phase I and Phase II groundwater data will not be combined.

Comment No. 18, Page 9-3 through and including 9.3.2.2, Statistics

RFI HLA will use two approaches to assess the presence of elevated levels of potentially hazardous materials in environmental media.

IDEM The information presented in these sections has been discussed on numerous occasions with various members of the Base Realignment and Closure Cleanup Team. During an Environmental Investigation meeting held in Chicago October 17-19, 1995, the Army stated that only a few details needed to be worked out regarding the ANOVA statistical method. The Army and EPA should finalize those details and the agreed upon approach should be incorporated into the Workplan.

As for the UTL approach it should follow the "Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities - Interim Final Guidance" (EPA, 1989) and the "Addendum to the Interim Final Guidance" (EPA, 1992).

IDEM will have the opportunity to evaluate and comment on the assumptions and recommendations made during the UTL and ANOVA statistical analysis.

Response

Comment noted.

Comment No. 19, Page 9-11, Section 9.3.1, second bullet

RFI Statistical analysis of background organic compound analytical results will not be performed because individual organic compounds were detected in less than 50 percent of the background samples analyzed.

IDEM Please incorporate a statement that indicates the Army will prepare a list(s) of all organic compounds that are found to be over their quantitation or detection limit for purposes of evaluation.

IDEM's Defense Environmental Restoration Program normally follows the IDEM Voluntary Cleanup Program guidelines as well as CERCLA and RCRA rules, regulations, and guidances. In this particular case IDEM staff have agreed to the use of the ANOVA technique in evaluating the similarities or differences of background/upgradient sample analytes to the site specific analytes.

The following refers to EPA RCRA's comments in a letter dated March 13, 1995 from Gale Hruska, Corrective Action Project Manager, to William Nelson:

The Army will report the background levels for those constituents with a 50% or greater frequency of non-detects, but will not perform a statistical analysis on the data. For constituents with less than 50% non-detect frequency, the Army will utilize the statistical methods presently in the report. If a comparison between the 50% or greater non-detect background levels and the respective constituent concentrations at particular solid waste management units are needed in the future, the Army and the U.S. EPA will, at that time, discuss how to best process the comparison.

Response

A bullet has been added to Section 9.3.1 indicating a list of organic compounds identified above the quantitation or detected limit has been added.

Comment No. 20, Page 9-13, Analysis of Variance

RFI The statistical populations defined using the visual inspection of box plots will be confirmed using the Kruskal-Wallis non-parametric ANOVA technique.

IDEM The ANOVA method should be used to evaluate the data using its own procedures and assumptions. This method should be conducted independent of the Upper Tolerance Limit approach. The purpose of using the ANOVA method is not to confirm the visual inspections of the box plots. The results of each of the statistical methods may or may not confirm previous assumptions.

Response

The respective Upper Tolerance Limit and the Analysis of Variance statistical evaluation procedures to compare investigative site data to background data will be performed independently of one another. The Kruskal-Wallis non-parametric ANOVA is a separate analysis of variance that can be used to help assess how the background data should be grouped for statistical analysis. For example this approach may be used to evaluate whether surface soil data should be grouped by soil association.

Comment No. 21, Page 9-14, 1st partial paragraph

IDEM The agencies may ask the Army to provide copies of the box plots, histograms and probability plots for review.

Response

The Histograms and Probability Plots explanation in Section 9.3.1.1 has been revised to indicate that histograms, and probability plots and other graphics will be provided if requested by the regulatory agencies.

Comment No. 22, Page 9-14, Shapiro-Wilk Test for Normality

IDEM The data should be tested for normality initially. Using histograms and probability plots to assume the distribution may not be accepted as suggested in the section discussing Histograms and Probability Plots.

Response

The Shapiro-Wilk test for normality will be conducted in concert with the histograms and probability plots. The combination of the graphical and numerical approaches is intended to more fully define the appropriate distribution than if any one of these tests were performed alone.

Comment No. 23, Page 9-15, Section 9.3.1.2, last sentence

RFI Analytes detected in investigative site samples that exceed their respective UTL will be identified as chemicals of concern.

IDEM For the purpose of the base closure being handled under CERCLA regulations and transfer of Army property, chemicals of concern will be determined upon evaluation of the ANOVA method. Please incorporate a sentence to indicate this.

Response

Text has been added to Section 9.3.2.2 indicating that "For the purposes of the Environmental Investigation proceeding under CERCLA for the transfer of Army property to other ownership, chemicals of concern will be assessed based on evaluation of results of the ANOVA statistical analysis."

Comment No. 24, Page 9-15 and 9-16, Section 9.3.2

IDEM This section should be revised according to discussions and agreements reached between EPA and the Army.

Response

Section 9.3.2, Statistical Method for Analytical Data Evaluation Using Analysis of Variance, has been revised according to discussions and agreements reached among EPA, the Army, and IDEM.

Comment No. 25, Page 9-16, Section 9.3.2.1, 1st paragraph

RFI The background data set will consist of the background data used previously for the UTL evaluation.

IDEM See IDEM comment #17.

Response

The background data set will consist of a combination of background soil data for samples collected during the Phase I and Phase II investigations. Upgradient groundwater data from Phase I and Phase II investigation groundwater samples will not be combined. Background soil data will be evaluated prior to combining the data. Because of changes in the analytical methods required by IDEM and the EPA Region V CERCLA program, some differences in Phase I and Phase II background data are anticipated.

Comment No. 26, Page 10-6, 2nd paragraph

RFI During USACE performance audits, approximately 20% of all project-specific analytical lots available at the laboratory will be examined.

IDEM The performance audits should include 100% of the analytical lots.

Response

Section 10.2.1.3 of the QAPjP has been revised. USAEC will not perform laboratory audits, but will rely on HLA's audits and subsequent audit reports. However, examination of 100 percent of the analytical lots during laboratory audits is not feasible. The laboratory audits coupled with the 100 percent data validation will ensure that the data quality meets the Phase II RFI DQOs.

Comment No. 27, Table 1.2

RFI "Summary of Phase I RCRA Facility Investigation (RFI)"

Background soil and groundwater samples were analyzed for Semivolatile Organic Compounds (SVOCs), Total Metals, Pesticides/Polychlorinated biphenyls (PCBs), Herbicides, Ammonia/Nitrates, and Landfill Parameters.

IDEM VOCs analysis should have been included for background samples. Any future background samples taken need to include all of the above listed analytes including VOCs.

Response

The Phase I subsurface-soil and groundwater samples were analyzed for VOCs. The Phase I background surface-soil samples were not analyzed for VOCs because of the tendency for VOCs to volatilize from surface soil.

The Phase II EI background soil samples will be analyzed only for metals because, at the direction of EPA and IDEM, background screening of investigative samples will be limited to metals.

Groundwater samples will be collected from monitoring wells on the upgradient boundary of the FBH property. Analytical results from the analysis of the upgradient groundwater samples will not be used for a statistical evaluation of background groundwater quality but may be used in a qualitative evaluation of groundwater samples collected from downgradient locations. Upgradient groundwater samples will be analyzed for VOCs, SVOCs, total and dissolved TAL metals including cyanide, TCL pesticides/PCBs, herbicides, and landfill parameters.

Comment No. 28, Tables 3.1 - 3.7

IDEM The document needs to be changed to use the Contract Lab Program's (CLP) Low Concentration Statement of Work (SOW) to be sure that the quantitation limit is below the MCLs and/or other legal limits for water.

Some of the tables will need to be revised using the lower quantitation/detection limits. Additional parameters are listed in the Low Concentration Statement of Work. Check the list of analytes in each of the tables and add analytes as required.

Response

The analytical methods for water have been revised to the CLP SOW methods (OLC01.0 and ILC01.0) for low concentration water analyses. Tables 3.1, 3.2, 3.3, and 3.5 have been updated with the lower CRQLs and CRDLs for VOCs, SVOCs, pesticides/PCBs, and metals, respectively. However, the MCLs for several compounds including benzo(a)anthracene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, dibromochloropropene, indeno(1,2,3-cd)pyrene and PCBs are lower than their respective CRQLs for the low concentration water analyses.

Comment No. 29, Table 3.1 - 3.7

RFI Water Quantitation Limits

IDEM The quantitation limits listed for water are unacceptable. Revise the list and incorporate the Region V Model QAPP Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Residential Well Water Samples. The quantitation limits must be below the MCLs or other legal limits.

Response

The analytical methods for water have been revised to the CLP SOW methods (OLC01.0 and ILC01.0) for low concentration water analyses, and the tables have been updated with the lower CRQLs and CRDLs from the methods. Because the CRQLs and CRDLs are specific to the respective CLP analytical methods, there is little flexibility afforded the laboratory to change these limits. Consequently, the CRQLs or CRDLs are, as identified in the response to Comment No. 28, exceed regulatory legal limits for some constituents.

Comment No. 30, Table 3.7

IDEM The table should indicate the Contract Required Detection Limits for soils.

Cyanide is missing from the list, please add cyanide to the list.

There is no table listing the CRDL's for the Inorganic Analyte List (TAL) and quantitation/detection limits for water. Please include this in a table. This table should list the analytical method.

Response

Cyanide has been added to the list of inorganic analytes.

Table 3.7 (current Table 3.5) has been revised to include CRDLs for water. The applicable CLP SOW does not provide CRDLs for soil. Therefore, the appropriate CRDLs were calculated from the sample preparation equations provided in the soil method relative to the CRDLs for water. Table 3.5 has also been updated to reflect the calculated CRDLs for soils.

Comment No. 31, Tables 3.1-3.7

IDEM Add a footnote or column that indicates the reference source of the quantitation limits and the analytical method.

Response

Footnotes referencing the appropriate methods and sources of the analyte lists and detection limits will be provided in Tables 3.1 through 3.7.

Comment No. 32, Table 7.1

IDEM Add the following footnote for herbicides: "SW-846 8000 will be used for general calibration and QC (as stated on page 7.1)."

Response

A footnote that "SW-846 Method 8000 will be used for general calibration and QC" has been added to Table 7.1.

**U.S. ENVIRONMENTAL PROTECTION AGENCY REGION V
COMMENTS (DATED OCTOBER 30, 1995) ON THE DRAFT QUALITY ASSURANCE
PROJECT PLAN FOR PHASE II RCRA FACILITY INVESTIGATION
FORT BENJAMIN HARRISON, MARION COUNTY, INDIANA
June 1995**

GENERAL COMMENT

The QAPP and TSP are integrally related documents which reference each other. The TSP should be compared to the QAPP by the U.S. Army to ensure that there are no remaining inconsistencies between the requirements of the two documents. Copies of both documents need to be available at the field site to members of the field crews for reference during sampling operations.

Response

The Quality Assurance Project Plan (QAPP) and the Technical Sampling Plan (TSP) have been reviewed to identify and resolve inconsistencies between the two documents. However, if the EPA is aware of any specific inconsistencies between the two documents, the inconsistencies should be identified for the Army. Copies of both documents will be available at Fort Benjamin Harrison for field personnel during the Phase II RFI field program.

SPECIFIC COMMENTS

Comment No. 1, Page 1, Signature Page

Please add the following names to this page: Karen Mason-Smith, United States Environmental Protection Agency, Superfund Remedial Project Manager and Richard Blume-Weaver, Fort Benjamin Harrison BRAC Environmental Coordinator (BEC). Please change Denise Boone's information to read as follows: United States Environmental Protection Agency, Superfund Chemist. (See Attachment A)

Response

The signature page has been revised as requested.

Comment No. 2, Page 1-4, Section 1.1

The text describes three areas related to background levels of metals, PAHs, and pesticides, that may need future investigation but these issues do not appear to be explicitly addressed in Section 1.2. Why are they not included in this investigation, and where will they be addressed.

Response

Background levels of metals in soil will be addressed during the Phase II Environmental Investigation with the collection of additional background surface-soil and subsurface-soil samples for metals analysis. Analytical results from these additional soil samples will be used to help evaluate investigative site data from the Phase II EI and RFI. The Phase II background samples will not be analyzed for polynuclear aromatic hydrocarbons (PAHs) or pesticides. These compounds are likely to be present in soil at FBH because of the urban and industrial setting of FBH. However, IDEM and the EPA have requested that organic compounds not be included in the statistical evaluation of ambient analyte concentrations in soil. Therefore, additional analysis of background samples for organic compounds has not been proposed.

Comment No. 3, Page 1-7, Section 1.5.1, Specify Objectives and Associated Tasks

Please specify the field and laboratory analysis associated with each Data Quality Objective (DQO) Level such as, DQO Level I for HNu, pH, temperature, and conductivity. This section describes the DQO levels for the sampling by media (groundwater, soil, and PCB screening). This summary would be more helpful if it were presented in a Table listing the specific site, the samples to be taken by media in that area, and the planned analyses and data uses for each analysis being performed. If such a table already exists in the TSP it should be referenced here.

Response

Brief summaries of the field or laboratory analyses associated with each Data Quality Objective (DQO) Level have been added to each bulleted DQO level description in Section 1.5. Section 1.5.1 will be revised to include Table 1.4. This table provides a summary, by site and by medium, of the proposed Phase II RFI sampling program, and the respective analyses, analytical methods, DQOs, and health-based target levels.

Comment No. 4, Figure 2.1 and Page 2-1, Section 2.0 Project Organization and Responsibility

Neither Fort Benjamin Harrison staff (Richard Blume-Weaver, BEC), Lorraine Wright and Manuella Johnson/IDEM, nor Karen Mason-Smith/U.S. EPA Region 5 Superfund are included in this organization chart. They should be included. (See the revised attached Figure 2.1, Attachment B and Attachment C). Please add Karen Mason-Smith as BRAC team (BCT) member with the overall responsibility of providing oversight to the CERCLA environmental investigations, overall responsibility of the review of base closure activities (including the review of "finding of suitability to lease" (FOSL) and "finding of suitability to transfer" (FOST) documents) and to review the QAPjP.

Response

The Quality Assurance Organization Structure chart (Figure 2.1) and accompanying text (Section 2.2) have been revised to include the BRAC Cleanup Team (BCT) and IDEM's Manuella Johnson.

Comment No. 5, Section 2.4, Laboratory Responsibilities

Provide the laboratory's address.

Response

Section 4.0 has been revised to include contact information (address and phone number) for the analytical laboratory.

Comment No. 6, Section 3.2, Accuracy, Precision, and Sensitivity of Analysis

The accuracy and precision QC criteria for herbicide analyses is reference to the SW-846 method, but the criteria is not stipulated. Therefore, furnish the laboratory specific criteria.

Response

The laboratory provided their current accuracy, precision, and sensitivity QC criteria for the analytical method in the herbicide SOP. The QC criteria are updated periodically by the laboratory, based on the

results of the laboratory's ongoing QC program, and at the time of analysis, may vary slightly from the information presented in the SOP. The herbicide, total petroleum hydrocarbons, cation exchange capacity, total organic carbon and polychlorinated dioxins and furans SOPs are provided in Appendix A.

Comment No. 7, Page 3-3, Section 3.3

Why is a completeness standard of 80% chosen? It is more typical to specify 90-95% completeness, which should be achievable within the QA/QC requirements of this document. Please provide additional explanation of the low expected completeness.

Response

The completeness standard has been revised to 90 percent.

Comment No. 8, Page 4-4, Section 4.3, Sample Containers, Preservatives and Volume Requirements

If containers are to be prepared in accordance with the EPA procedures, reference the guidance; if not, provide the procedures.

Response

The following information has been added to Section 4.3 of the QAPjP. The laboratory will provide sample bottles and vials purchased from commercial sources and certified by the supplier as analyte-free for project target compounds. Sample container preparation procedures are described in Table 4.1

Comment No. 9, Tables 4.1 Sample Container, Preservation, and Holding Times for Subsurface Soil Sample Analyses

- a. *Identify "PCBs" and "Pest/PCBs".*
- b. *Correct the holding times for SVOCs, Pest/PCBs, and Herbicides to "14 days until extraction and 40 days after extraction".*
- c. *Correct the holding time for Dioxins/furans to "30 days until extraction and 45 days after extraction".*

Response

Table 4.2 (formerly Table 4.1) has been revised to identify pesticides/PCBs. Separate identification of PCBs is unnecessary because the same sample container, preservative, and holding time requirements apply to PCBs as to pesticides/PCBs.

The holding times for SVOCs, pesticides/PCBs, and herbicides have been revised to "14 days until extraction and 40 days after extraction." The holding times for dioxins/furans have been revised to "30 days until extraction and 45 days after extraction."

Comment No. 10, Tables 4.2 Sample Container, Preservation, and Holding Times for Surface and Excavated Soil Sample Analyses

- a. *Correct the holding times for SVOCs, Pest/PCBs, and Herbicides to "14 days until extraction and 40 days after extraction".*
- b. *Correct the holding time for Dioxins/furans to "30 days until extraction and 45 days after extraction".*

Response

Table 4.3 (formerly Table 4.2) holding times for SVOCs, pesticides/PCBs, and herbicides have been revised to "14 days until extraction and 40 days after extraction." The holding times for dioxins/furans have been revised to "30 days until extraction and 45 days after extraction."

Comment No. 7, Table 4.3 Sample Container, Preservation, and Holding Times for Groundwater Sample Analyses

- a. *Identify "PCBs" and "Pest/PCBs".*
- b. *Correct the holding time for Dioxins/furans to "30 days until extraction and 45 days after extraction".*

Response

Table 4.4 (formerly Table 4.3) has been revised to identify pesticides/PCBs. Separate identification of PCBs is unnecessary because the same sample container, preservative, and holding time requirements apply to PCBs as to pesticides/PCBs. The holding times for dioxins/furans have been revised to 30 days until extraction and 45 days after extraction.

Comment No. 8, Section 5.1.3 Field Logbooks and Documentation

Describe the sample identification numbering system and provide at least two examples of the system.

Response

The sample identification numbering system is based on requirements of the USAEC's database. The sample identification number is unique for each sampling location. The sample identification number identifies the RFI site, the sample medium, and sampling location at the site. More than one sample collected at a sampling location is uniquely identified by the site type or depth fields listed on the COC forms, sample labels, and sample tags. Example sample numbers used for the Phase I RFI are described below.

SM011SB007

SB007 refers to soil boring (SB) number 007 collected at Solid Waste Management Unit #FBH11.

SM017SS003

SM017 refers to surface-soil (SS) sample number 003 collected at Solid Waste Management Unit #FBH17

A description of the sample numbering system is provided in the Phase II RFI TSP, Data Management Plan (Appendix B), Section 4.4.

Comment No. 9, Section 6

The QAPP references the CLP methods for analysis of the samples, yet the methods are not cited for calibration of laboratory instruments. Since these methods contain specific requirements for calibration of CLP analyses, please reference them in this section.

Response

Section 6.2 has been revised to reference Table 7.1, which provides a specific listing of the methods to be used for sample analysis. Instruments will be calibrated in accordance with the procedures provided in these methods.

Comment No. 10, Page 8-1, Section 8.1.2

The text mentions an optical difference of 0.2 between replicate standards. What is the significance of this difference? How does it relate to concentration differences in the samples. Additional information on the significance of this difference should be included in this section if it is to be used as a means of assessing the quality of PCB screening results.

Response

Method calibration and quality control documentation is an integral part of the EnSys immunoassay tests. Based on the manufacturer's instructions, a valid test is indicated when the magnitude of the displayed number is 0.20 or less. Test runs resulting in a number greater than 0.20 will be repeated to ensure valid conclusions.

Comment No. 11, Page 9-11, Section 9.3.1

The contents of the statistical discussion need to be consistent with the ongoing discussions between Fort Benjamin Harrison, IDEM, and U.S. EPA. In light of those separate, ongoing discussions, this section was not reviewed.

Response

Comment noted.

Comment No. 12, Appendix A Standard Operating Procedure for Gas Chromatographic Analysis of Chlorinated Herbicides in Water and Soil, Section 8.0 Quality Control

Provide the acceptance criteria for the method blank, standard matrix spike, sample matrix spikes sample matrix spike duplicate, and samples spiked with surrogate.

Response

The laboratory has provided their current accuracy, precision, and sensitivity QC criteria for the herbicide analytical method (Herbicide SOP, Appendix A). The information provided includes criteria for method blank, standard matrix spike, sample matrix spikes, sample matrix spike duplicate, and

surrogate recovery. The QC criteria are updated periodically by the laboratory, based on the results of the laboratory's ongoing QC program, and at the time of sample analysis, may vary slightly from the information presented in the SOP.

**RESPONSE TO INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
COMMENTS (DATED MARCH 15, 1996) REGARDING FORT BENJAMIN HARRISON
DRAFT PHASE II RESOURCE CONSERVATION RECOVERY ACT
FACILITY INVESTIGATION QUALITY ASSURANCE PROJECT PLAN,
JUNE 1995**

Army Revised Response, January 22, 1996:

Army Response to Comment No. 6

Table 1.4 has been added to Section 1 of the Phase II RFI QAPP. This table provides a summary, by site, by medium of the proposed Phase II RFI sampling program, by the respective analyses, DQOs, and risk-based action levels. (An example table (Table I) is attached (Attachment 1) to this response package.)

IDEM Staff Comment

See IDEM staff response to the Army's transmittal of Risk-Based Action Level Table (Reference 5).

Response

Comment noted.

Army Response to Comment No. 17

The Army submitted a description of the proposed Phase II background sampling program to IDEM and EPA on October 13, 1995. The submittal included a general description of proposed surface and subsurface soil background sampling and upgradient groundwater sampling for regulatory agency review and comment. Although intended to be part of the EI, the Phase II background sampling program will be implemented concurrently with the Phase II RFI field program.

The October 13, 1995, plan indicates that the analytical results from those Phase I background soil samples previously accepted by IDEM will be combined with the Phase II background soil sample results to define background at FBH. The respective Phase I and Phase II background soil data will be evaluated to assess whether the data are similar before the data sets are combined. Because of changes in the analytical methods required by IDEM and the EPA Region V CERCLA program, some differences in the Phase I and Phase II soil background results may occur. Phase I and Phase II groundwater data will not be combined.

IDEM Staff Comment

See IDEM Staff response to the Army's proposed Phase II Environmental background sampling program (Reference 1C).

Response

Comment noted.

**Army's January 17, 1996 Transmittal of the Risk-Based Action Level Table and Table 2:
Rise-Based Action Level Table:**

On January 22, 1996, IDEM staff received the Army's Risk-Based Action Table. Please include this table in the RFI TSP for reference purposes only. The table should be referred to as "Program specific goals and levels used to assess data," not "Risk-based action level table."

An IDEM chemist noted that there should be an entry placed in the table for total polychlorobiphenyls. The individual PCBs do not have maximum contaminant levels, but there is a total PCB MCL of 0.5 ppb (Drinking Water Regulations and Health Advisories by the Office of Water, U.S. EPA, Washington, D.C. May 1995). Please add this to the table.

Enclosed, please find a revised version of the State of Indiana's Voluntary Remediation Program Resource Guide. Several Tier II goals have been revised. This guide may be used to update the table. Also enclosed please find, a copy of the U.S. EPA Region IX Preliminary Remediation Goals. Please add Region IX PRGs to the table.

Please send IDEM staff a copy of the EPA "Soil Screening Guidance" referenced.

Response

As agreed during the March 22, 1996, conference call, the table formerly entitled "Risk-Based Action Level Table" has been renamed "Summary of Health-Based Target Levels." The table has been revised to include the PCB maximum contaminant level (MCL) of 0.5 $\mu\text{g/l}$. Additionally, the IDEM Tier II Goals listed in the table have been compared to the revised Tier II Goals listed in the October 1995 *State of Indiana's Voluntary Remediation Program Resource Guide* and updated as appropriate. The Army has also added EPA Region IX Preliminary Remediation Goals to the table. The revised "Summary of Health-Based Target Levels" table was resubmitted to the Army and regulatory agencies on April 12, 1996.

Table 2

Site Identification:

Two of the sites, the former sewage treatment plant (SWMUnit #11) and the former sanitary waste incinerator (SWMU #17) are missing from the table. Please add them.

Response

Table 2 included with the January 22, 1996, revised draft response to comments on the Draft Phase II Resource Conservation and Recovery Act Facility Investigation Work Plan, Fort Benjamin Harrison, Marion County, Indiana, was provided in response to an EPA comment as an example of the proposed table to elicit comments regarding the table's format. It was not intended to be complete. A complete version of the table is provided in the Final QAPP for the Phase II RFI (Table 1.4).

Analysis:

The analysis should match what was agreed to in previous meetings and correspondence (i.e., fax list of analytes).

Response

The list of analytes shown in the table is consistent with the list of analytes agreed to in previous meetings, conference calls, and correspondence.

Data Use:

Data Quality Levels are appropriate.

Response

Comment noted.

Regulatory Levels:

The Army requested that the agencies review and comment on the Regulatory Levels column. The column should be labeled "levels to assist in determining the necessary levels of analytical precision and accuracy" or "levels used to establish analytic methods," not "regulatory levels." MCL's are regulatory levels, but DQLs are not.

Action levels will be determined based on the outcome of the health and ecological risk assessments.

The Region V Data Quality Levels CANNOT be used as action levels or regulatory levels. RCRA DQLs are used to establish analytical methods. U.S. EPA Region V issued a memo (28 June 1994) regarding RCRA Corrective Action Guidance and Human Health Data Quality Levels for RFI Projects. This memo presented Data Quality Levels, as Region V RCRA Permitting Branch guidance, to assist in determining the necessary levels of analytical precision and accuracy. The Region V RCRA Permitting Branch Data Quality Levels, July 1994, attached to the memo, presented the RCRA DQLs and stated that the DQLs are NOT intended to represent any of the following:

- 1. "Cleanup" levels for purposed of RCRA remediation;*
- 2. "Reporting limits" for purposes of submitting RFI data to the U.S. EPA;*
- 3. Anticipated detection limits, expect in cases where a laboratory-specific method detection limit closely corresponds with a published Practical Quantitation Limit or Estimated Quantitation Limit;*
- 4. Predetermined basis for RFI "Phase II" screening levels;*
- 5. Form the basis for determining whether the "No Further Action" alternative is justified, without completion of a risk assessment or comparison to site-specific action levels;*
- 6. Ecotoxicity thresholds, such that contaminant concentrations not exceeding these levels could be assumed to be protective of ecological resources; and*
- 7. Contaminant detection levels sufficient to assess risk to local ecological resources.*

If the Army wishes to use the MCLs and DQLs as their site specific action levels, risk assessments (health and ecological) must still be performed on chemical analytes found to be above background (or equal to or below the MCLs and DQLs). If there are ecological concerns, an evaluation must be made as to whether the MCLs are protective.

IDEM staff recommend using Ecotox Threshold benchmark values, established for surface water and sediment, for screening purposes only. If surface water and sediment analyte concentrations are below the Ecotox Threshold benchmark values, no action will be required. If the surface water and sediment concentrations are above the Ecotox values the Army will need to conduct a risk assessment. Enclosed, please find a copy of ECO Update Publication 9345.0-12FSI (January 1996), listing Ecotox Thresholds. Ecotox Thresholds are defined as media specific contaminant concentrations above which there is a sufficient concern regarding adverse ecological effects to warrant further site investigation. ETs are meant to be used for screening purposes only; they are not regulatory criteria, site-specific cleanup standards, or remediation goals.

IDEM staff recommend using the 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) level indicated in the EPA Region IX Preliminary Remediation Goals and the Draft Toxicity Equivalency Factors (TEF) for chlorinated dibenzo-p-dioxins (CDDs) and chlorinated dibenzofuran (CDFs). Enclosed, please find the table from the Region IX, EPA Preliminary Remediation Goals indicating the PRG for 2,3,7,8-tetrachlorodibenzo-p-dioxin

(TCDD). *The Draft Toxicity Equivalency Factors are to be used for screening purposes only. If the Army has additional information it is planning to use, please submit it for review.*

The Army requested the use of SW-846, method 8290, for dioxins/furans instead of method 8280 or the CLP method, because it uses a high resolution GC/high resolution mass spectrometer, has lower detection levels (parts per trillion ranges), and it looks at specific isomers for sites. The Army is planning to use method 8290 to test dioxins and furans on site SM25c. IDEM staff approve the use of the dioxin/furan method 8290 for the RFI sites. Specific isomers can be detected using this method.

If sample concentrations meet the following criteria for dioxins and furans, then they could be screened out and excluded from the Risk Assessment:

dioxin/furan levels equal to or below the Region IX Preliminary Goals (9/1/95 or current version), and individual dioxin/furan isomers equal to or below the concentrations calculated by their Toxicity Equivalency Factors.

Response

This comment was discussed with the regulatory agencies on March 22, 1996. As a result of those discussions, the column heading formerly entitled "Regulatory Level" has been retitled "Health Based Target Levels."

DQLs were included in this table for reference purposes and were not intended to be interpreted as representing site-specific action levels. Site-specific action levels will be based on the outcome of a site-specific risk assessment (health and ecological) at the sites where a CMS and risk assessment are required by Region V EPA (RCRA Program).

IDEM correctly noted in their comment that DQLs do not represent cleanup levels, reporting limits, detection limits, screening levels, no further action levels, ecotoxicity thresholds, or contaminant detection levels sufficient to assess risk to local ecological resources. However, the EPA Region V DQL April 1994 summary document states that "DQLs are intended to guide facilities in the direction of health-based, target levels to which analytical data may be compared in the future." The Army included DQL values in Table 2 to indicate the type of risk-based (health-based) target levels to which Phase II RFI investigative sample analytical results will be compared in the future.

Inclusion of MCLs and DQLs in Table 2 was not intended to indicate that these values should be used for site-specific action levels. As discussed above, site-specific action levels will be based on the outcome of a site-specific risk assessment (health and ecological) at those RFI sites where a CMS and risk assessment are requested by the Region V EPA (RCRA Program). MCLs are based, in part, on human health concerns and should not be used to evaluate ecological concerns.

The Army appreciates IDEM's recommendation that Ecotox Threshold Benchmark Values may be used for the screening of surface-water and sediment data. However, surface-water and sediment samples will not be collected during the Phase II RFI. (Surface-water and sediment samples may be collected as part of the Phase II EI.)

Region IX PRGs for 2,3,7,8-tetrachlorodibenzo-p-dioxin(TCDD) have been included in the "Summary of Health Based Target Levels" table. The Region III risk-based values and MCLs for TCDD are also included in the table. If other dioxin/furan congeners are identified, the Army will use the Draft Toxicity Equivalency Factors to evaluate these congeners.

The Army did not request the use of SW-846 Method 8290 for dioxins/furan analysis of Phase II RFI samples. Previously, the EPA and IDEM have requested that when possible, the Army use only CLP methods for analysis of Phase II RFI samples. The Army complied with this request and proposed the

Appendix D

CLP dioxin/furan method for analysis of Phase II RFI samples. During the March 22, 1996, conference call among the Army, EPA, IDEM, and their consultants, IDEM agreed to provide a written request that Phase II RFI dioxin/furan sample be analyzed using SW846 8290 method. In response to this written comment, the Army will analyze the Phase II RFI samples for dioxin/furans using the EPA's SW-846 Method 8290.

The Army acknowledges IDEM's comment regarding screening out sites using adjusted dioxin/furan concentrations. However, under the RCRA Corrective Action program, the Army has agreed to perform risk assessment at those sites requested by EPA Region V (RCRA Program).

Army Request for re-evaluation of Agency request to check 100 percent of analytical calculations during data validation:

The Army requested that staff check with IDEM chemistry section regarding a previous comment pertaining to data validation.

Billy Crawford, IDEM chemist, consulted IDEM's Quality Assurance Officer (Manuela Johnson) regarding the 100% quality assurance/quality control validation. Both chemists agree that the 20% manual recalculation of analytical calculations is acceptable.

Response

Comment noted.